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Results of LLNL's Participation in the 16th OPCW Proficiency Test

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This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

Results of LLNL's Participation in the 16th OPCW Proficiency Test

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In the summer and fall of 2004, LLNL scientists prepared samples for the 16th official Organisation for the Prohibition of Chemical Weapons (OPCW) Proficiency Test.

Our report of this effort is attached.

This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

ORGANISATION FOR THE PROHIBITION OF CHEMICAL WEAPONS

Report of the Sixteenth Official OPCW Proficiency Test

Part I: Sample Preparation

Laboratory code: Sample Prep Lab

Total number of pages: 128[1]

[1] Total number of pages including cover page and all attachments

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Armando Alcaraz, Principle Investigator
Hugh Gregg, co-PI, reporting
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Robert Maxwell, NMR

Packaging and Shipping
Marina Chiarappa-Zucca
Sarah Chinn
Peter Nunes
Phil Pagoria
Tom Tillotson
Rich Whipple
LLNL shipping staff

1 Introduction

The Sixteenth Official OPCW Proficiency Test started in October 2004. The samples were prepared by scientists affiliated with the Forensic Science Center at the Lawrence Livermore National Laboratory in Livermore, California, USA. The work was funded by the US Department of Energy.

The test scenario and the spiking and background chemicals were discussed and agreed in advance with the OPCW. The samples were prepared in accordance with "Work Instruction for the Preparation of Test Samples for OPCW Proficiency Tests" (Document No.: QDOC/LAB/WI/PT2).

The preparation of the test samples and their analysis are described in this report.

2 Test Scenario

A State Party has presented in accordance with Article IX of the Convention an inspection request for a challenge inspection. The Executive Council did not decide against this request, and the Director-General, in accordance with Part X of the Verification Annex, sent a challenge inspection team to a certain facility that has been accused of producing chemical warfare agents.

The facility is described by the inspection team as being a chemical research plant with well-equipped analytical and synthesis laboratories. The inspection team collected one sample inside the analytical laboratory from a container labeled "Organic Waste". Another sample was collected outside a synthesis laboratory from a large unlabeled plastic container, "Liquid". The inspection team collected a third sample, "Soil", outside the facility's chemical loading/unloading area. The inspection team did not analyze the samples on-site and forwarded them directly for off-site analysis. In addition, the inspection team could not collect corresponding blanks and used pure organic solvents and pre-cleaned soil similar to that found at the facility.

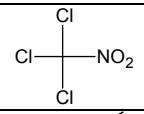
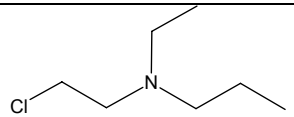
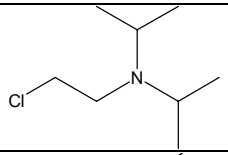
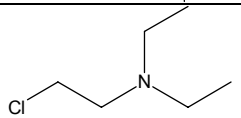
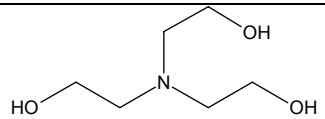
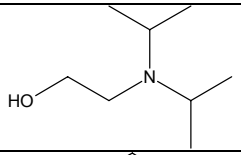
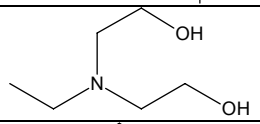
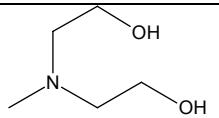
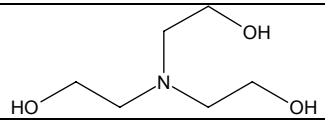
Please analyze the samples for the presence of **any Scheduled chemicals and/or their degradation/reaction products**, taking into account the characteristics of the samples.

3 List of Scheduled Chemicals

The majority of the chemicals used in this Proficiency Test were obtained commercially. Several spiked chemicals (both scheduled and unscheduled) were synthesized in-house. The purities of the scheduled chemicals were determined by NMR (^{1}H and $^{13}C\{^{1}H\}$) and those results are presented in Part II, *Purity Checks*, of this report.

The Scheduled chemicals spiked into the three matricies are presented in the table on the next page.

List of Spiking Chemicals for the Sixteenth Official OPCW Proficiency Test

Sample code	No	Compound name	CAS number	Spiking Chemical Structure	Schedule number
O	A	Trichloronitromethane	76-06-2		3.A.04
O	B	2-(N-Ethyl-N-propylamino)ethylchloride			2.B.10
O	C	2-(N,N-Diisopropylamino)ethylchloride	96-79-7		2.B.10
O	D	2-(N,N-Diethylamino)ethylchloride	100-35-6		2.B.10
L	E	Triethanolamine	102-71-6		3.B.17
L	G	2-(N,N-Diisopropylamino)ethanol	96-80-0		2.B.11
S	H	Ethyldiethanolamine	139-87-7		3.B.15
S	I	Methyldiethanolamine	105-59-9		3.B.16
S	J	Triethanolamine	102-71-6		3.B.17

4 Sample Preparation

Several preliminary stability studies were carried out in the months preceding the 16th Official Proficiency Test. These studies were used to down-select the list of scheduled chemicals used for spiking and helped to determine stable amounts of materials and containers. Based on these studies, and in consultation with OPCW, three sample matricies were spiked as shown below.

4.1 Organic sample (O)

The following chemicals constituted the Organic sample.

Component	Origin/purity	Concentration (mg/L)
Trichloronitromethane	In-house synthesis	30
2-(N-Ethyl-N-propylamino)ethylchloride	In-house synthesis[1]	10
2-(N,N-Diisopropylamino)ethylchloride	Aldrich[1], 97%	6
2-(N,N-Diethylamino)ethylchloride	Aldrich[1], 99%	30
Diesel fuel	Local gas station[1]	200
Carbon tetrachloride	J.T. Baker, 100%	20
2,4-Dinitro-t-butylbenzene	In-house synthesis, 97%	40
2,4-Dinitrotoluene	Eastman Kodak	20
2-Nitroaniline	Eastman Kodak	20
Hexane	Fluka, min. 99.5%	Solvent

[1]The preparation of the chlorides and diesel fuel cleanup are presented in section 4.4.

4.2 Liquid sample (L)

The following chemicals constituted the Liquid sample.

Component	Origin/purity	Concentration (mg/L)
Triethanolamine	Aldrich, 98%	500
2-(N,N-Diisopropylamino)ethanol	Aldrich, 99%	30
2-(N,N-Diethylamino)ethanol	Aldrich, 98%	20
2-(N,N-Dimethylamino)ethanol	Aldrich, 99%	20
2-(N-Butyl-N-methylamino)ethanol	In house synthesis	30
2,6-Dinitroaniline	Aldrich, 97%	20
2-Nitroaniline	Eastman Kodak	20
Pyridine	Sigma Aldrich, 99.9%	20
Poly(ethyleneglycol), M_n 200	Aldrich	200
Methanol	Riedel-deHaën, Chromasolv®, min 99.9%	Solvent

4.3 Soil sample (S)

The following chemicals constituted the Soil sample.

Component	Origin/purity	Concentration (mg/kg)
Ethyldiethanolamine	Aldrich, 98%	20
Methyldiethanolamine	Aldrich, 99.9+%	20
Triethanolamine	Aldrich, 98%	20
Diesel fuel	Local gas station[1]	200
Caffeine	Aldrich, 99%	40
Nicotine	Sigma, 99%	40
Malation	Chem Service, 99.2%	40
Tributylamine	EM, 98%	40
Tripentylamine	EM, 98%	40
1,3-Dinitrobenzene	Aldrich, 97%	40
2,4-Dinitrotoluene	Eastman Kodak	20
Dichloromethane	Aldrich, 99.9%	(dried)
Sand, purified	J.T. Baker	Matrix

[1]The preparation of the chlorides and diesel fuel cleanup are presented in section 4.4.

4.4 Chemical preparation

The three scheduled chlorine-containing amines were purchased or synthesized as their hydrochloride salts. These salts were converted to the chloride form by adding 1.0 mL of aqueous sodium hydrogen carbonate to a known amount of material and extracting the formed chloride into three serial aliquots of 1.0 mL of hexane. The resulting hexane extracts were dried using a column of anhydrous sodium sulfate. To estimate the resulting solution concentrations, it was assumed that 100% conversions and recoveries of the chlorides into the hexane were accomplished.

Diesel fuel was obtained from a local gas station and washed prior to use. To wash the fuel, 100 mL of fuel was placed in a 500 mL round-bottomed flask and 100 mL of concentrated sulfuric acid was added. The mixture was stirred vigorously for 2 hours. The two phases were placed in a separatory funnel and the sulfuric acid layer was discarded. The fuel layer was washed sequentially with 20 mL water, 10 mL of a 5% solution of sodium hydrogen carbonate, and 20 mL of water. The washed fuel was dried over anhydrous sodium sulfate and finally dried over anhydrous sodium carbonate, yielding a clear, colorless liquid.

The remaining chemicals were obtained or synthesized neat and ready to use.

4.5 Protocols for preparing test samples

The soil samples and blank soil samples were prepared on October 6, 2004 and the two liquid samples and their corresponding blanks were prepared on October 7, 2004. Two staff members of the OPCW laboratory witnessed the sample preparation process. A total of 40 sets of samples and their corresponding blanks were made. Seventeen sets of samples and their corresponding blanks

were sent to laboratories that were planning to participate in the 16th OPCW Proficiency Test, two sets of samples and blanks were sent to the evaluation laboratory, and two sets of samples and blanks were sent to the OPCW laboratory. Three sets of samples and blanks, chosen randomly by the OPCW staff members, were used by the preparation laboratory for stability testing. An additional set was used by the preparation laboratory for qualitative analysis. The remaining sample sets were retained by the sample preparation laboratory for contingencies.

4.6 Organic sample (Code O) and Organic sample blank (Code OB) preparation

Hexane was added to a 500 mL, glass, solvent-rinsed, volumetric flask. Next, 125 microliters of diesel fuel was added to the hexane. Stock solutions of individual test chemicals were made in hexane and ranged in concentration from 1-30 mg/mL. A sufficient volume of each stock solution was added to the hexane solution to yield the desired analyte concentrations, listed in the previous section. The hexane solution was brought to a final volume of 500 mL and analyzed by GC/MS to verify that the solution was of the expected composition.

Neat hexane was used as the organic solvent blank. The hexane was analyzed by GC/MS to verify that it contained none of the added test chemicals.

4.7 Liquid sample (Code L) and Liquid sample blank (Code LB) preparation

Methanol was added to a glass, solvent-rinsed, 2.5 L jug. Next, 250 microliters of poly(ethyleneglycol) was added to the methanol. Stock solutions of individual test chemicals were made in methanol and ranged in concentration from 5-50 mg/mL. A sufficient volume of each stock solution was added to the methanol solution to yield the desired analyte concentrations, listed in the previous section. The methanol solution was brought to a final volume of 1.0 L and analyzed by GC/MS to verify that the solution was of the expected composition.

Neat methanol was used as the organic solvent blank. The methanol was analyzed by GC/MS to verify that it contained none of the added test chemicals.

4.8 Soil sample (Code S) and Soil sample blank (Code SB) preparation

To make the soil samples, the test chemicals were first added to dichloromethane. The resulting dichloromethane solution was added to the sand and allowed to evaporate, leaving the test chemicals on the soil.

Dichloromethane was added into a glass, solvent-rinsed, 2.5 L jug. Next, 625 microliters of diesel fuel was added to the dichloromethane. Stock solutions of individual test chemicals were made in dichloromethane and ranged in concentration from 10-20 mg/mL. A sufficient volume of each stock solution was added to the dichloromethane solution to yield the desired analyte concentrations, listed in the previous section. The dichloromethane solution was brought to a final volume of 1.25 L and analyzed by GC/MS to verify that the solution was of the expected composition.

Into each of five, solvent-rinsed, 1-L beakers were placed 500 g sand. Into each beaker containing sand, 250 mL of the above dichloromethane solution were added. The resulting mixture was stirred with a metal spatula and placed in a chemical fume hood. The dichloromethane was allowed to evaporate overnight, leaving the test chemicals on the sand. When the sand was dry, it was again

mixed in the beaker and transferred into an empty bottle, which had previously contained clean sand. The sand contained in all of the beakers (2500 g total) was transferred to the bottle. The bottle containing spiked sand was then placed on a roller and homogenized for approximately 5 minutes. The resulting spiked sand was then sampled, extracted, derivitized and analyzed by GC/MS to verify that it was of the expected composition.

Blank samples were prepared by exposing 2500g of sand to 1.25 L of dichloromethane. The blank sand was prepared in a manner identical to that of the spiked soil, with the exception that no test chemicals were added. The resulting blank sand was sampled, extracted, derivitized and analyzed by GC/MS to verify that it was free of test chemicals.

4.9 Standards

Two sets of standards were prepared, one each for the OPCW laboratory and the evaluation laboratory. These standards were prepared by dilution from the stock solutions in the range of 1 to 2 mg/mL. A set of vials of each standard, along with a description, was shipped a few days after the samples were shipped.

5 Packaging and Transportation

The sample preparation laboratory received the list of participating laboratories on 28 September 2004 and contacted them on several occasions to confirm delivery addresses and preferred shippers. It was gratifying that most laboratories responded promptly, however there were significant communication issues with several laboratories.

5.1 Announcement of Sample Dispatch

Annex A contains a copy of the fax and/or e-mail detailing the sample dispatch information.

5.2 Packaging

10 mL of the organic solution (hexane) and corresponding blank were pipetted into 20-mL vials (pre-cleaned borosilicate glass) and capped (Teflon-lined, silicon-backed seal). The caps were tightened, taped with a thin strip of duct tape followed by the application of a tamper indicating seal. These bottles were labeled and placed inside of plastic bottles with absorbent material. These secondary bottles were sealed with a heat-shrink plastic seal.

18 mL of the liquid solution (methanol) and corresponding blank were pipetted into 20-mL vials (pre-cleaned borosilicate glass) and capped (Teflon-lined, silicon-backed seal). The caps were tightened, taped with a thin strip of duct tape followed by the application of a tamper indicating seal. These bottles were labeled and placed inside of plastic bottles with absorbent material. These secondary bottles were sealed with a heat-shrink plastic seal.

50 grams of the soil and the soil blank were weighed into 2-oz. clear glass jar and sealed with caps (Teflon-bonded to polypropylene foam). The caps were tightened, taped with a thin strip of duct tape followed by the application of a tamper indicating seal. These bottles were labeled and sealed inside of a heat-sealable plastic bag.

All six containers (O, OB, L, LB, S, SB) were placed inside of a foam-lined cardboard box. Each box was accompanied by a letter (Annex B). The shipping information was affixed to the exterior of the box.

5.3 Confirmation of Dispatch

The LLNL shipping department took custody of the samples and provided tracking numbers. Participating laboratories were notified of the carrier and tracking number (see Annex C for an example). Due to multiple transportation restrictions, multiple carriers had to be used.

5.4 Sample Receipt

At least 10 of the 17 laboratories received the samples within one week of shipping. A summary of the shipping and receipt information is provided in Annex D.

6 Analysis of Samples

The three sample matrices were analyzed both quantitatively and qualitatively.

6.1 Quantitative Analysis

Each of the three matricies required different sample work up procedures.

6.1.1 Analytes in Organic Solution

Analysis of spiked chemicals in the organic solution was performed by direct injection of one microliter of sample solution into the GC/MS (EI). Quantitation was performed using extracted ion chromatograms and using heptadecane (present as a component of the diesel fuel background) as an internal standard. A three point calibration curve (10 to 40 mg/L) was prepared fresh for each sampling day.

m/z EICs used for quanitation

Cmpd #	Compound name	EIC m/z
A	Trichloronitromethane	117
B	2-(N-Ethyl-N-propylamino)ethylchloride	120
C	2-(N,N-Diisopropylamino)ethylchloride	106
D	2-(N,N-Diethylamino)ethylchloride	86

6.1.2 Analytes in Liquid Solution

Analysis of spiked chemicals in the organic solution was performed by direct injection of one microliter of sample solution (compound E was diluted with methanol before injection) into the GC/MS (EI). Quantitation was performed using extracted ion chromatograms and using PEG200 (present as a component added to the background) as an internal standard. A three point calibration

curve (for compound E: 50 to 200 mg/L; for compound G: 10 to 40 mg/L) was prepared fresh for each sampling day.

m/z EICs used for quantitation

Cmpd #	Compound name	EIC m/z
E	Triethanolamine	118
G	2-(N,N-Diisopropylamino)ethanol amine	114

6.1.3 Analytes in Soil Sample

Analysis of spiked chemicals in the soil sample was performed by extraction, derivatization, and direct injection of one microliter of sample solution into the GC/MS (EI). Quantitation was performed using extracted ion chromatograms using heptadecane (present as a component of the diesel fuel background) as an internal standard. A three point calibration curve (10 to 40 mg/L) was prepared fresh for each sampling day and was derivatized in the same manner and at the same time as the sample extracts.

The extraction of the sand was accomplished by sonicating 2.0 g of sand with 2.0 mL of methanol for 15 minutes. The resulting solution was centrifuged for 3-5 minutes and 500 µL of the clarified extract were transferred to a 4-mL vial. The extract was evaporated to dryness with a gentle stream of nitrogen and reconstituted immediately with 200 µL of N, O-Bis(trimethylsilyl)trifluoroacetamide (BSTFA). The resulting solution was sealed and heated at 60ºC for 30 minutes to allow derivatization of the three amines.

m/z EICs used for quanitation

Cmpd #	Compound name	EIC m/z
H	Ethyldiethanolamine	174
I	Methyldiethanolamine	160
J	Triethanolamine	262

6.1.4 Quantitative Analysis Results

Chemical A: Trichloronitromethane (mg/L)

Sample	**Day 0**	**Day 7**	**Day 11**	**Day 15**	**Day 21**	**Day 29**	**Day 40**
O/25	30	25	26	32	26	26	26
O/35	33	30	24	30	26	25	27
O/40	33	28	29	30	26	26	31
Average	32	28	26	31	26	26	28
%RSD	5.4%	9.1%	9.6%	3.8%	1.0%	0.9%	8.0%

Chemical B: 2-(N-Ethyl-N-propylamino)ethylchloride (mg/L)

Sample	Day 0	Day 7	Day 11	Day 15	Day 21	Day 29	Day 40
O/25	10.4	10.6	10.7	11.0	10.4	10.7	10.2
O/35	10.9	11.5	10.2	10.4	10.9	9.5	10.7
O/40	11.3	11.6	11.4	10.7	11.4	10.1	11.4
Average	10.9	11.2	10.8	10.7	10.9	10.1	10.8
%RSD	4.2%	4.7%	5.4%	2.9%	4.5%	6.0%	5.7%

Chemical C: 2-(N,N-Diisopropylamino)ethylchloride (mg/L)

Sample	Day 0	Day 7	Day 11	Day 15	Day 21	Day 29	Day 40
O/25	5.3	5.2	5.4	5.2	4.8	5.8	5.2
O/35	6.0	5.6	5.2	5.0	5.1	5.1	5.4
O/40	6.5	5.7	5.8	5.2	5.2	5.5	5.7
Average	5.9	5.5	5.5	5.1	5.1	5.5	5.4
%RSD	9.9%	4.2%	5.3%	2.1%	3.9%	5.8%	4.8%

Chemical D: 2-(N,N-Diethylamino)ethylchloride (mg/L)

Sample	Day 0	Day 7	Day 11	Day 15	Day 21	Day 29	Day 40
O/25	24	23	23	25	25	23	24
O/35	25	25	22	24	26	21	25
O/40	25	25	24	24	27	22	26
Average	25	24	23	24	26	22	25
%RSD	3.8%	5.9%	4.9%	3.8%	4.6%	4.7%	4.6%

Chemical E: Triethanolamine (mg/L)

Sample	Day 0	Day 7	Day 11	Day 15	Day 21	Day 29	Day 40
O/25	433	374	384	418	426	516	500
O/35	487	490	477	409	476	564	557
O/40	530	516	461	482	506	512	579
Average	483	460	441	436	469	531	545
%RSD	10%	16%	11%	9.0%	8.6%	5.5%	7.5%

Chemical G: 2-(N,N-Diisopropylamino)ethanol (mg/L)

Sample	Day 0	Day 7	Day 11	Day 15	Day 21	Day 29	Day 40
O/25	30	28	29	33	33	28	26
O/35	32	29	25	32	31	26	30
O/40	33	30	27	31	31	28	29
Average	32	29	27	32	32	28	28
%RSD	4.6%	3.2%	7.6%	4.0%	4.3%	4.5%	7.7%

Chemical H: Ethyldiethanolamine (mg/kg)

Sample	Day 0	Day 8	Day 12	Day 16	Day 22	Day 30	Day 40
O/25	10.3	8.6	7.5	10.9	7.9	10.0	9.3
O/35	11.6	9.6	10.9	9.7	8.1	10.0	10.2
O/40	10.2	10.3	10.8	11.8	7.3	10.4	10.8
Average	10.7	9.5	9.7	10.8	7.8	10.2	10.1
%RSD	7.2%	8.8%	20%	9.7%	5.2%	2.3%	7.6%

Chemical I: Methyldiethanolamine (mg/kg)

Sample	Day 0	Day 8	Day 12	Day 16	Day 22	Day 30	Day 40
O/25	7.5	7.6	7.4	6.6	5.3	7.0	7.3
O/35	9.3	7.9	8.2	6.2	5.5	6.8	7.7
O/40	8.3	7.7	7.7	7.8	4.5	7.2	7.7
Average	8.4	7.7	7.8	6.9	5.1	7.0	7.6
%RSD	11%	2.1%	5.4%	12%	10%	3.1%	3.0%

Chemical J: Triethanolamine (mg/kg)

Sample	Day 0	Day 8	Day 12	Day 16	Day 22	Day 30	Day 40
O/25	8.7	10.4	9.2	11.3	8.1	12.5	12.7
O/35	9.6	10.9	9.5	8.7	8.0	12.3	13.0
O/40	6.6	13.0	8.9	12.1	6.9	12.9	12.8
Average	8.3	11.4	9.2	10.7	7.7	12.6	12.8
%RSD	19%	12%	3.3%	17%	8.2%	2.2%	1.1%

6.2 Qualitative Analysis

The results of the qualitative analysis are presented in Part III, *Qualitative Analysis*, of this report. That section of the report is documented using the standard forms for reporting results of an Official OPCW Proficiency Test.

The Organic samples were analyzed directly by GC/MS EI & CI. The Liquid samples were analyzed after derivitization with BSTFA to form the trimethylsilyl derivative and then analyzed by GC/MS EI & CI. The Soil samples were first extracted with methanol, derivitized with BSTFA to form the trimethylsilyl derivative and then analyzed by GC/MS EI & CI.

Detailed procedures are described in Part III of this report.

Annex A: Dispatch Announcement

October 6, 2004

University of California
Lawrence Livermore National Laboratory
7000 East Ave
Livermore, CA 94550

Date of Sample Dispatch for the Official 16th OPCW Proficiency Test

Dear Colleagues,

The samples for the 16th Official OPCW Proficiency Test will be dispatched from our laboratory on **8 October 2004**. FedEx or other air fright carrier will deliver the packages. The airway bill numbers will be released on the day of sample dispatch. The test samples contain an organic sample, liquid sample, and soil with their corresponding blanks.

Please acknowledge the receipt of the fax or email to:

Fax: (925) 423-9014
Email: alcaraz1@llnl.gov

Good luck with the analysis.

Armando Alcaraz

Sincerely,
Armando Alcaraz
Forensic Science Center
Lawrence Livermore National Laboratory
Voice: (925) 423-6889
Fax: (925) 423-9014

Annex B: Letter Accompanying Package

October 8, 2004

University of California
Lawrence Livermore National Laboratory
7000 East Ave
Livermore, CA 94550

Participating Laboratories
16th Official OPCW Proficiency Test

TEST SAMPLES FOR THE 16TH OFFICIAL OPCW PROFICIENCY TEST

Dear Colleagues,

1. Please find enclosed samples for chemical analysis according to the information which you received from the OPCW regarding the 16th OPCW test scenario.
2. The package contains four vials sealed in plastic containers and two jars sealed in plastic wrap. The samples are an organic solvent, liquid, and sand with their corresponding blanks. They are labeled:

   Samples:
   The three samples coded are as follows:
   - O: Sample from container "Organic Waste"
   - L: Sample appearing to be an organic liquid
   - S: Soil from the facility's chemical loading/unloading area

   with their corresponding blanks
   - OB
   - LB
   - SB

   Your laboratory code is a two-digit number, which has been selected at random.

3. Please confirm the arrival date of the samples at your laboratory and their condition, by fax or email to:
   Mr. Stefan Mogl, OPCW Laboratory (Fax:+31 15 2840679, email: OPCWrij@worldonline.nl)

Regards,

Armando Alcaraz

Annex C: Dispatch Notification

The following is an example of e-mail or fax notification.

Dear Prof. Col József Fürész and Lt Gellért Karvaly,

Below is the web address and FedEx tracking number for your OPCW test samples:

http://www.fedex.com/cgibin/tracking?action=track&language=english&cntry_code=us&initial=x&tracknumbers=40090164874

FedEx Track numbers = 40090164874

Please acknowledge the receipt of the fax or email to:

Fax: (925) 423-9014
Email: alcaraz1@llnl.gov

Best regards,
Armando Alcaraz
Program Element Leader
Lawrence Livermore National Laboratory
Forensic Science Center, L-178
7000 East Ave
Livermore, CA 94550-9234
(925) 423-6889 phone
(925) 423-9014 fax

Annex D: Sample Receipt

Country	Name of laboratory and address	Air Freight Carrier	Airway bill number	Receipt package date
Austria	RD-ARWT/ABCUT Vorgartenstr. 225 A-1024 Wien Austria	FedEx	40090164815	13/10/2004
Belgium	DLD (Departement Laboratoria van Defensie) Kwartier Majoor Housiau Martelarenstraat 181 B-1800 Vilvoorde (Peutie) Belgium	FedEx	40090164841	12/10/2004
Czech Republic	Research Institute for Organic Syntheses Rybitvi 296 532 18 Pardubice 20 Czech Republic	FedEx	40090164852	12/10/2004
Denmark	Ministry of Defence, Emergency Management Agency, Chemical Division Universitetparken 2, Nørre Alle 67, 7th floor DK-2100 Copenhagen Denmark	FedEx	4009016505025	12/10/2004
Finland	Finnish Institute for Verification of the Chemical Weapons Convention (VERIFIN) P.O. Box 55 00014 University of Helsinki A.I.Virtasen aukio 1, 00560 Helsinki Finland	Air France	057-4146 8000	21/10/2004
France	Centre d'Etudes du Bouchet (CEB) Section Analyses Chimiques PO Box 3 91710 Vert-le-Petit France	FedEx	40090164863	11/10/2004

Hungary	Department of Toxicology Institute of Health Protection Hungarian Defence Forces 44 Róbert károly körút, Budapest, H-1134 P.O.Box 68, Budapest, H1555 Hungary	FedEx	40090164874	12/10/2004
India-IICT	Indian Institute of Chemical Technology Analytical Division Tarnaka Hyderabad-500 007 Andhra Pradesh India	Emirates Airlines	176-7000 2936	19/10/2004
India - VERTOX	Defence Research & Development Establishment VERTOX Laboratory Jhansi Road Gwalior 474002 India	Cathay Pacific Airways	160-3462 0622	19/10/2004
Malaysia	Department of chemistry Malaysia Ministry of Science, Technology and Innovation Jalan Sultan 46661 Petaling Jaya Malaysia	Cathay Pacific Airways	160-3462 0611	15/10/2004
Morocco	Laboratoire Officiel d'Analyses et de Recherches Chimiques (LOARC) 25, Rue Nichakra Rahal 20 000 Casablanca Morocco	Air France	057-4146 79996	10/10/2004*
Netherlands	TNO Prins Mauris Laboratory Lange Kleiweg 137 2288 GJ Rijswijk The Netherlands	FedEx	40090164911	12/10/2004
Romania	NBC & Ecological Defence Scientific Research Centre Sos. Oltenitei 225, sector 4 RO-75 6872 BUCHAREST Romania	Air France	057-4146 8011	01/11/2004

Russia CAL	Central Chemical Weapons Destruction Analytical Laboratory GosNIIOKhT (CAL) Shosse Entusiastov, 23 111024 Moscow Russian Federation	Air France	057-4146 8022	27/11/2004*
Spain	Fábrica Nacional "La Marañosa", Carretera San Martin de la Vega. Km. 10.5 San Martin de la Vega Madrid 28330 Spain	FedEx	40090164966	15/10/2004
Sweden	Swedish Defence Research Agency (FOI) Division of NBC Defence Cementvägen 20 SE-901 82 UMEÅ Sweden	FedEx	40090164970	14/10/2004
Ukraine	State Analytical Laboratory 61024 Kharkiv Petrovskogo St. 28 Ukraine	Lufthanza Cargo	020-7274 8712	01/11/2004*
UK-DSTL	Defence Science and Technology Laboratory, Porton Down, Salisbury Wiltshire, England SP4 0JQ	FedEx	40090164981 40090164756	11/10/2004 11/10/2004
OPCW Laboratory	OPCW Laboratory Heulweg 28-30 2288 GN Rijswijk The Netherlands	FedEx	40090165003 40090164760	12/11/2004 12/11/2004

* This date represents the date when the Proficiency Test samples arrived at the point-of-entry only. These laboratories did not respond to the preparation laboratory, so the date (if any) of receipt of the samples at their facility is unknown.

ORGANISATION FOR THE PROHIBITION OF CHEMICAL WEAPONS

Report of the Sixteenth Official OPCW Proficiency Test

Part II: Purity Checks

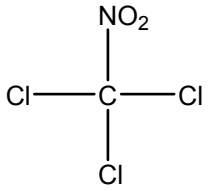
Laboratory code: Sample Prep Lab

TABLE OF CONTENTS

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** A

Aliquot codes: CW-CK-1-132-2	
Sample:	**Trichloronitromethane in CD_2Cl_2**

Compound structure:	

NMR Method name:	**^{1}H NMR**

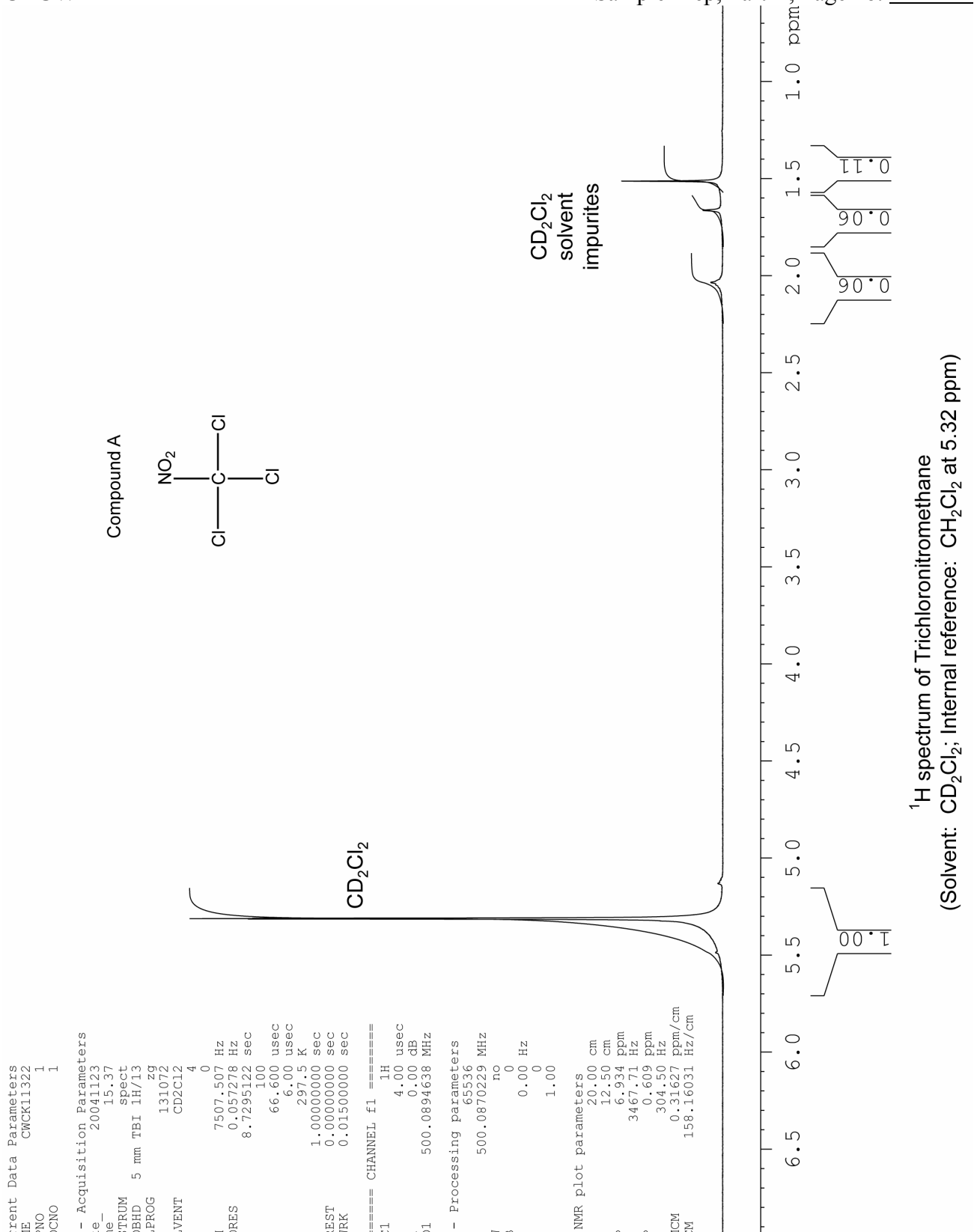
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	500.09 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis	☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code)	☐ Other:
☐ Standard addition:	Source :	
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.	

Experiments:	☒ ^{1}H ☐ ^{13}C{^{1}H} ☐ ^{19}F ☐ ^{31}P{^{1}H} ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
Interpretative comments	There are no protons in this sample. All peaks in ^{1}H NMR spectrum are due to the solvent and its impurities (confirmed by background NMR analysis of the solvent). Purity checks were performed with ^{13}C{^{1}H} analysis.			



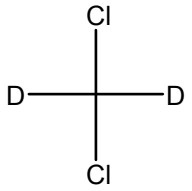
^{1}H spectrum of Trichloronitromethane

(Solvent: CD_2Cl_2; Internal reference: CH_2Cl_2 at 5.32 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: **Prep Lab** **Sample code(s):** **Purity Check** **Compound number:** **(solvent)**

Aliquot codes: CW-CK-1-132-1	
Sample:	**Solvent background CD_2Cl_2**

Compound structure:	

NMR Method name:	**^{1}H NMR**

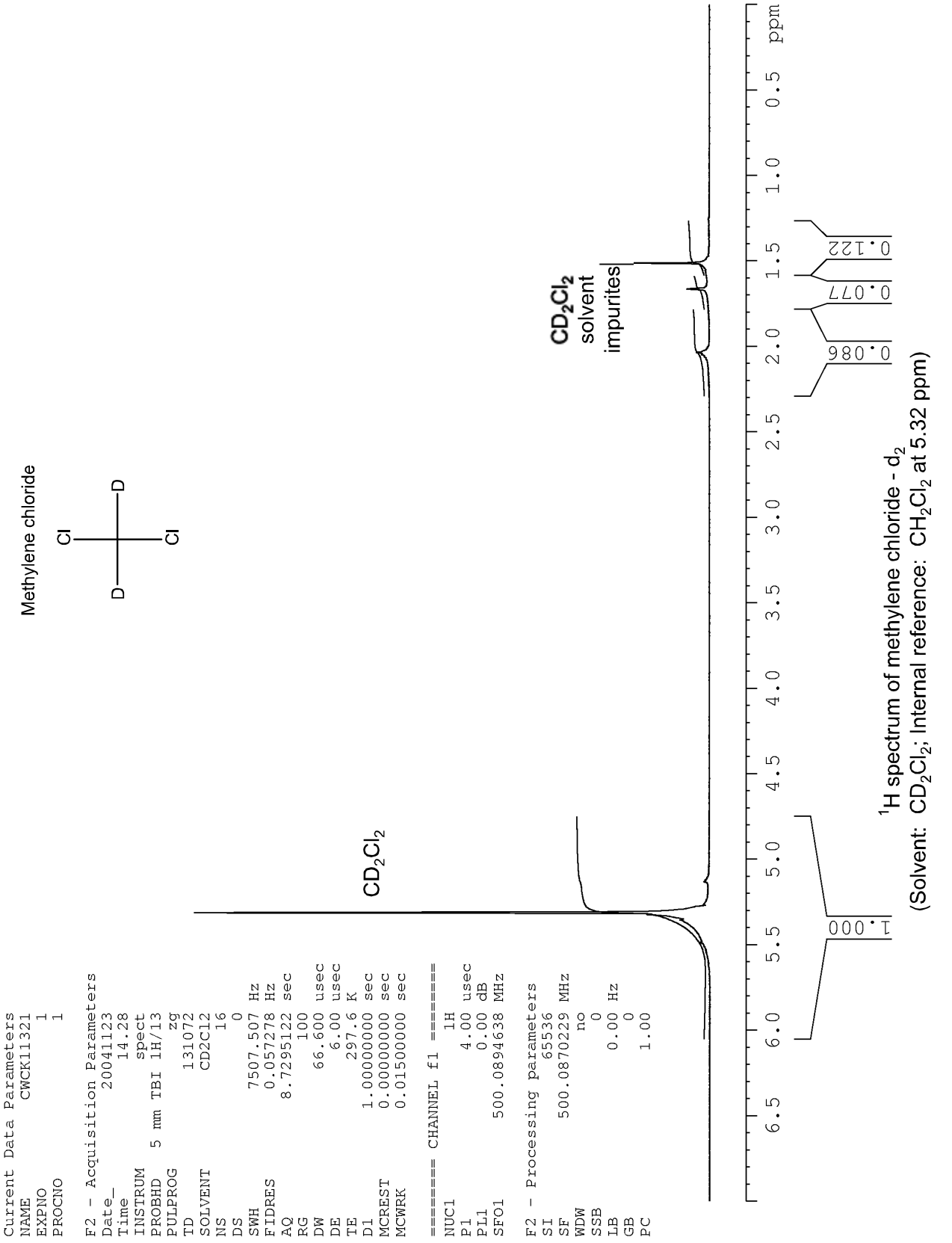
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	500.09 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code) ☐ Other:
☐ Standard addition:	Source :
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.

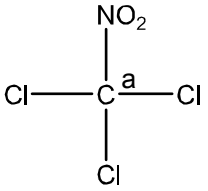
Experiments:	☒ ^{1}H ☐ ^{13}C{^{1}H} ☐ ^{19}F ☐ ^{31}P{^{1}H} ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
Interpretative comments	Background ^{1}H NMR of solvent confirming solvent impurities shown in ^{1}H NMR of sample A.			


Current Data Parameters
NAME CWCK11321
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041123
Time 14.28
INSTRUM spect
PROBHD 5 mm TBI 1H/13
PULPROG zg
TD 131072
SOLVENT CD2C12
NS 16
DS 0
SWH 7507.507 Hz
FIDRES 0.057278 Hz
AQ 8.7295122 sec
RG 100
DW 66.600 usec
DE 6.00 usec
TE 297.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 4.00 usec
PL1 0.00 dB
SFO1 500.0894638 MHz
F2 - Processing parameters
SI 65536
SF 500.0870229 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Methylene chloride
Cl
D
D
Cl
CD2Cl2
CD2Cl2
solvent
impurites
1.000
0.086
0.077
0.122
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1H spectrum of methylene chloride - d2
(Solvent: CD2Cl2; Internal reference: CH2Cl2 at 5.32 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** A

Aliquot codes: CW-CK-1-131-1	
Sample:	**Trichloronitromethane in CD_2Cl_2**

Compound structure:	

NMR Method name:	**$^{13}C\{^1H\}$ NMR**

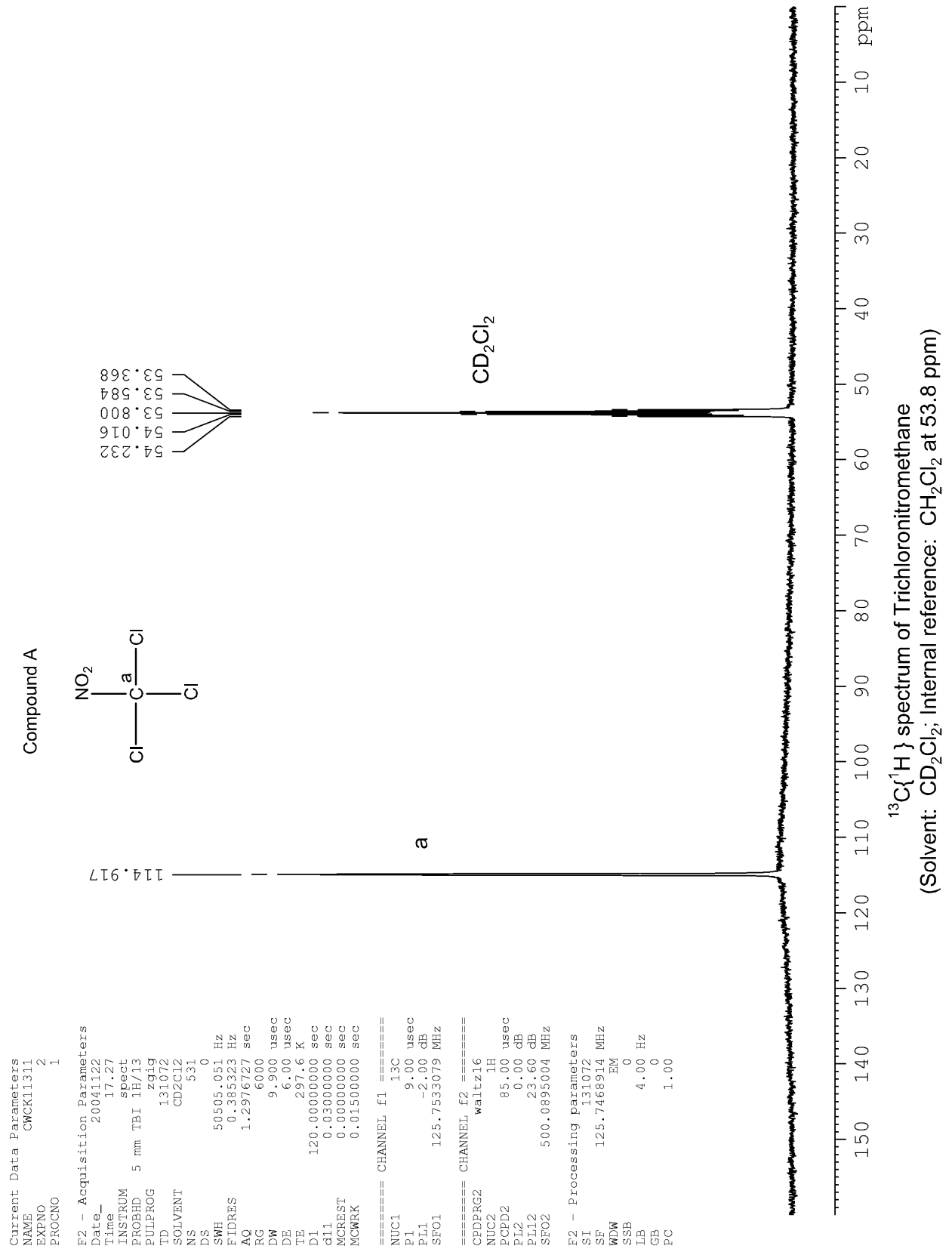
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	125.75 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CH_2Cl_2/CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code) ☐ Other:
☐ Standard addition:	Source :
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.

Experiments:	☐ ^1H ☒ $^{13}C\{^1H\}$ ☐ ^{19}F ☐ $^{31}P\{^1H\}$ ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
a	114.92 ppm			
Interpretative comments	Purity = 100%			

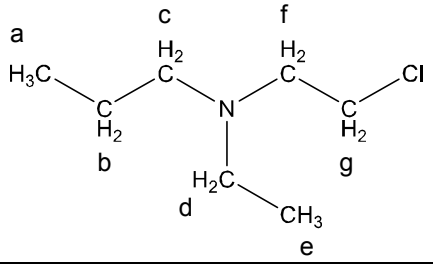


$^{13}C\{^{1}H\}$ spectrum of Trichloronitromethane
(Solvent: CD_2Cl_2; Internal reference: CH_2Cl_2 at 53.8 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** B

Aliquot codes: CW-CK-1-89-2	
Sample:	**2-(N-Ethyl-N-propylamino)ethylchloride in CH_2Cl_2/CD_2Cl_2**

Compound structure:	

NMR Method name:	**^{1}H NMR**

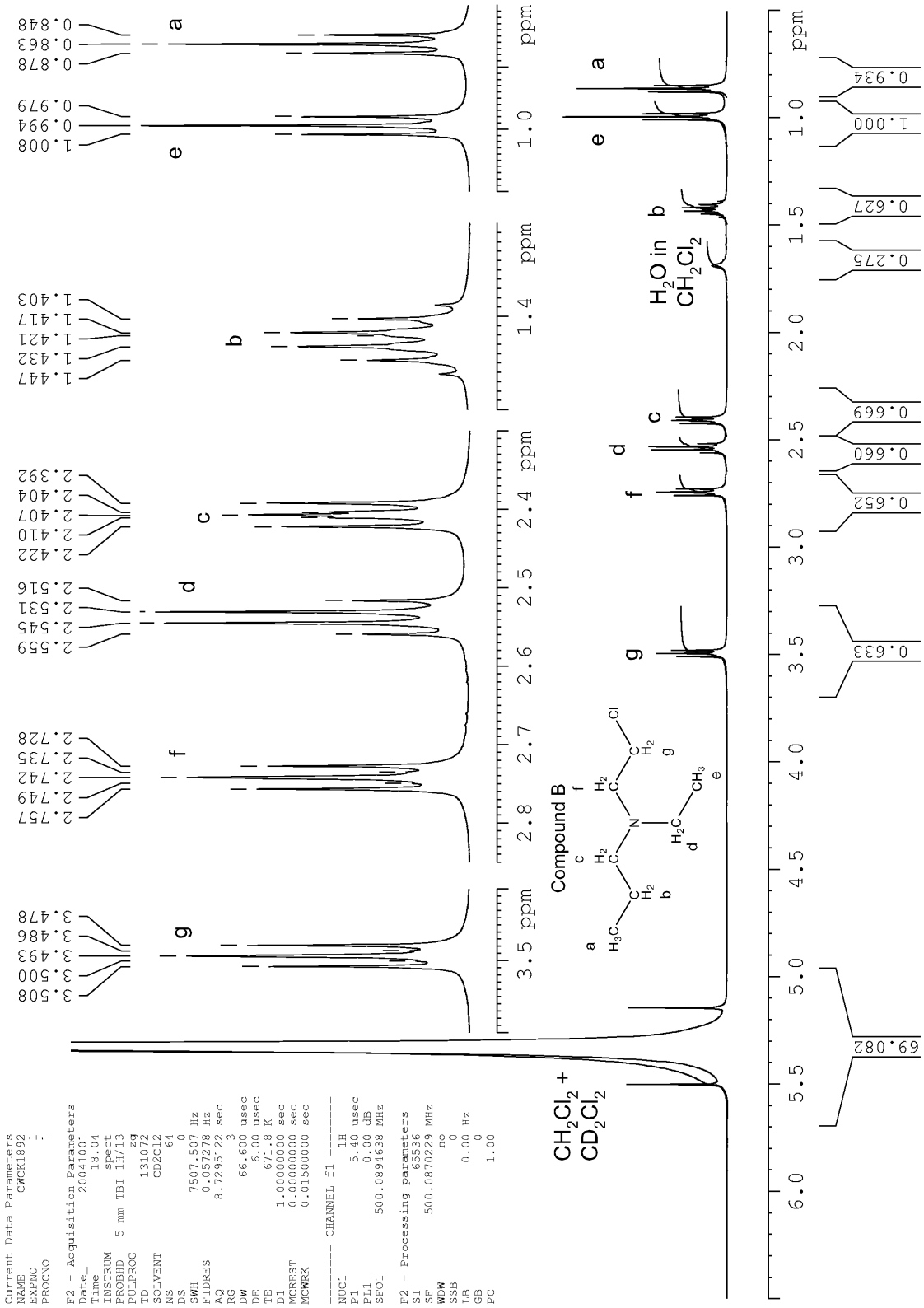
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	500.09 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CH_2Cl_2/CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code) ☐ Other:
☐ Standard addition:	Source :
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.

Experiments:	☒ ^{1}H ☐ ^{13}C{^{1}H} ☐ ^{19}F ☐ ^{31}P{^{1}H} ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
a	0.86		7.5 (b,a)	
e	0.99		7.1 (d,e)	
b	1.42		7.5 (a,b; c,b)	
c	2.41		7.5 (b,c)	
d	2.53		7.1 (e,d)	
f	2.74		7.5 (g,f)	
g	3.49		7.5 (f,g)	
Interpretative comments	Purity = 99.96%			

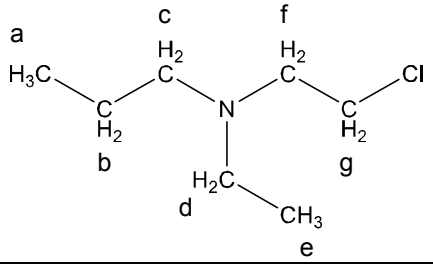


^{1}H spectrum of 2-(N-Ethyl-N-propylamino)ethylchloride
(Solvent: CH_2Cl_2 + CD_2Cl_2; External reference: CD_2Cl_2 at 5.32 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: **Prep Lab** **Sample code(s):** **Purity Check** **Compound number:** **B**

Aliquot codes: CW-CK-1-89-2	
Sample:	**2-(N-Ethyl-N-propylamino)ethylchloride in CH_2Cl_2/CD_2Cl_2**

Compound structure:	

NMR Method name:	**$^{13}C\{^1H\}$ NMR**

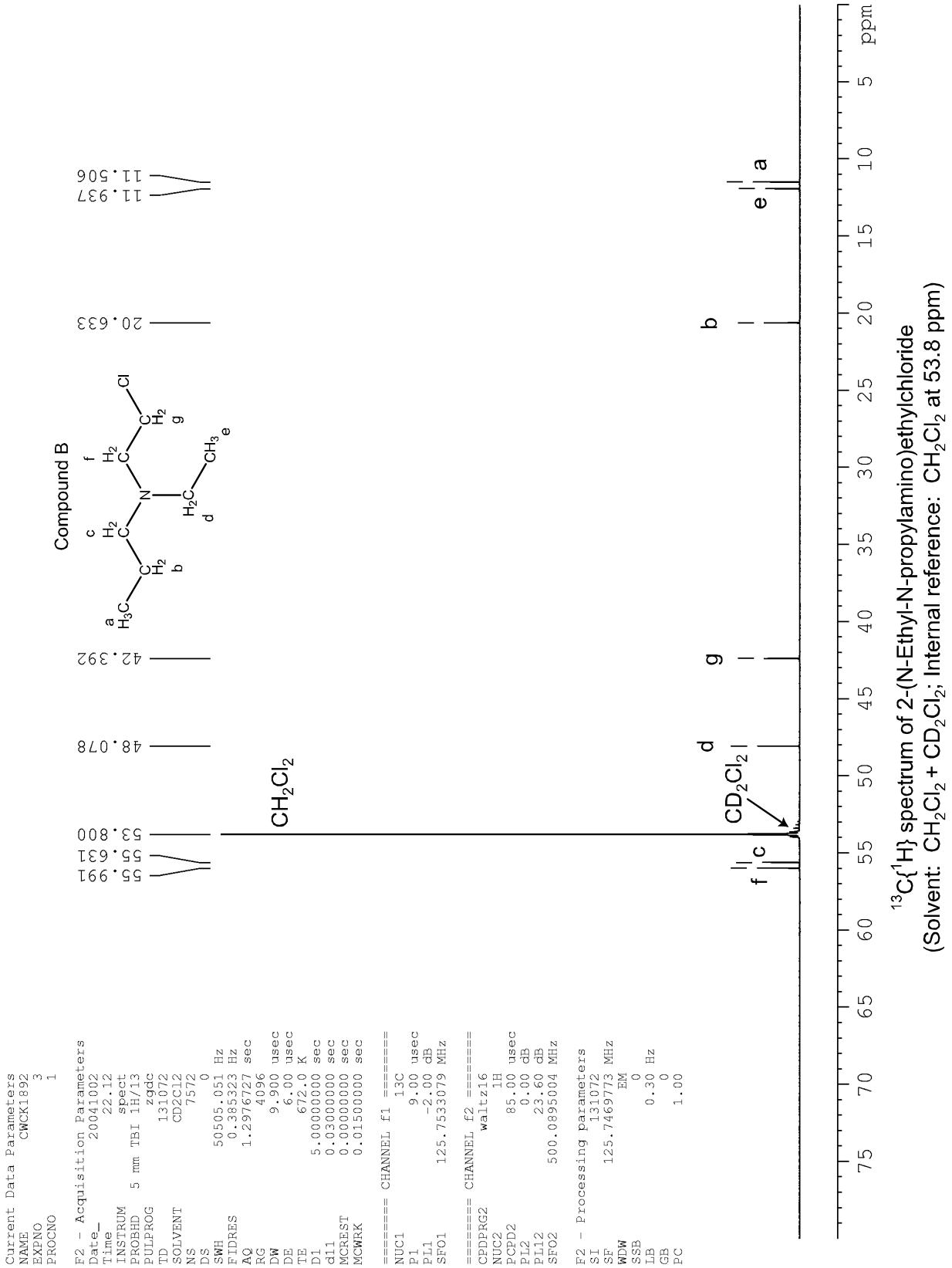
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	125.75 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CH_2Cl_2/CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code) ☐ Other:
☐ Standard addition:	Source :
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.

Experiments:	☐ ^1H ☒ $^{13}C\{^1H\}$ ☐ ^{19}F ☐ $^{31}P\{^1H\}$ ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
a	11.51			
e	11.94			
b	20.63			
g	42.39			
d	48.08			
c	55.63			
f	55.99			
Interpretative comments				

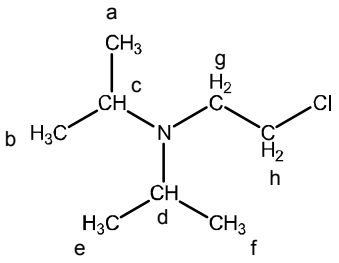


$^{13}C\{^1H\}$ spectrum of 2-(N-Ethyl-N-propylamino)ethylchloride
(Solvent: CH_2Cl_2 + CD_2Cl_2; Internal reference: CH_2Cl_2 at 53.8 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** C

Aliquot codes: CW-CK-1-131-2	
Sample:	**2-(N,N-diisopropylamino)ethylchloride in CD_2Cl_2**

Compound structure:	

NMR Method name:	**^{1}H NMR**

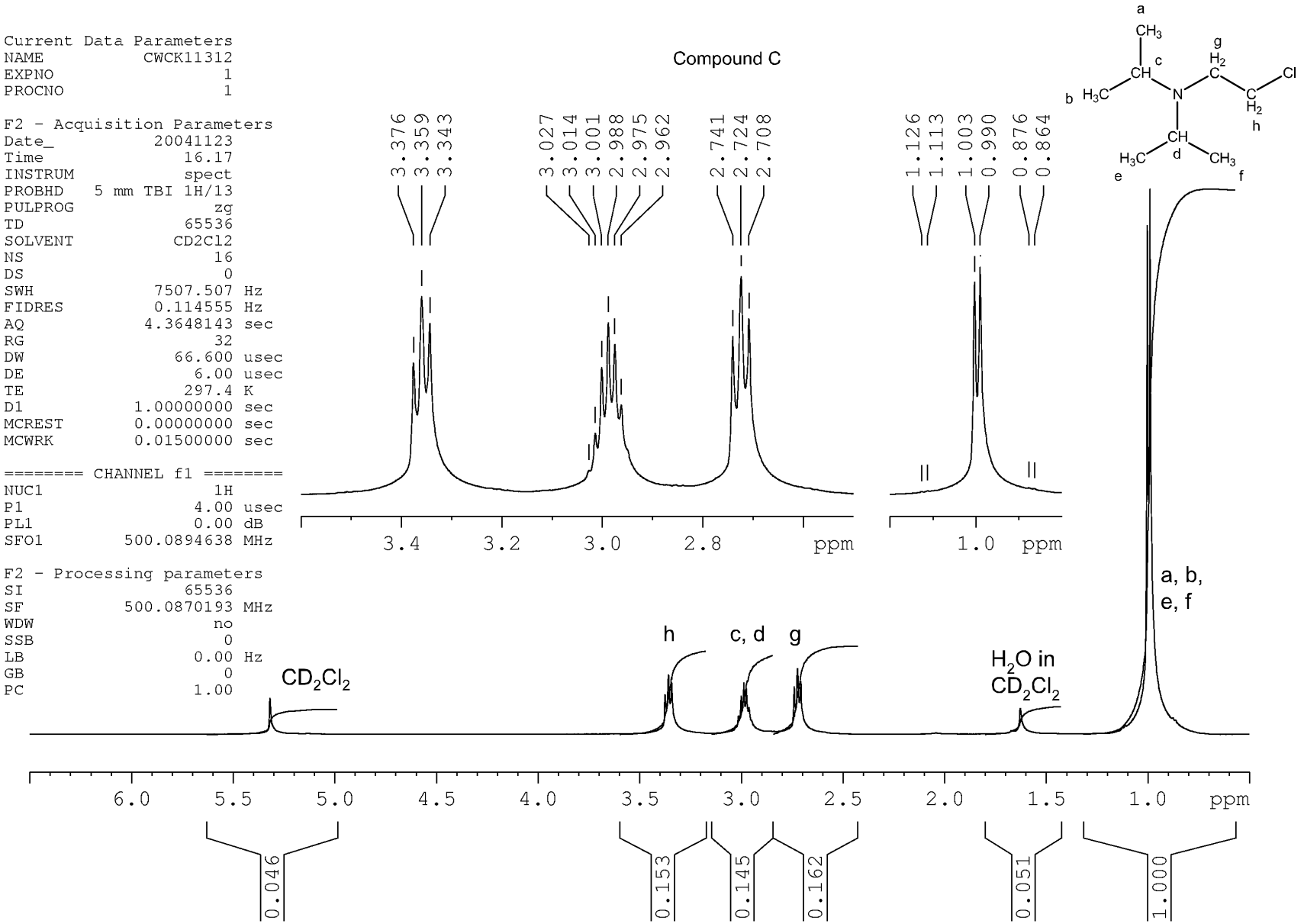
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	500.08 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code) ☐ Other:
☐ Standard addition:	Source :
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.

Experiments:	☒ ^{1}H ☐ ^{13}C{^{1}H} ☐ ^{19}F ☐ ^{31}P{^{1}H} ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
a,b,e,f	1.00, 0.99		6.5 (a,c; b,c; e,d; f;d)	
g	2.72		8.1 (g,h)	
c,d	2.99		6.5 (c,a; c,b; d,e; d,f)	
h	3.36		8.1 (h,g)	
Interpretative comments	Purity > 99.9%			

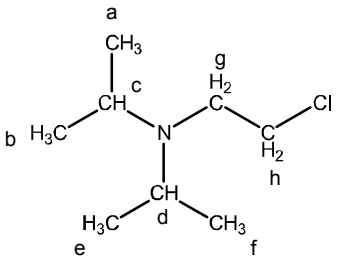


^{1}H spectrum of 2-(N,N-Diisopropylamino)ethylchloride
(Solvent: CD_2Cl_2; Internal reference: CH_2Cl_2 at 5.32 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** C

Aliquot codes: CW-CK-1-131-2	
Sample:	**2-(N,N-diisopropylamino)ethylchloride in CD_2Cl_2**

Compound structure:	

NMR Method name:	**$^{13}C\{^1H\}$ NMR**

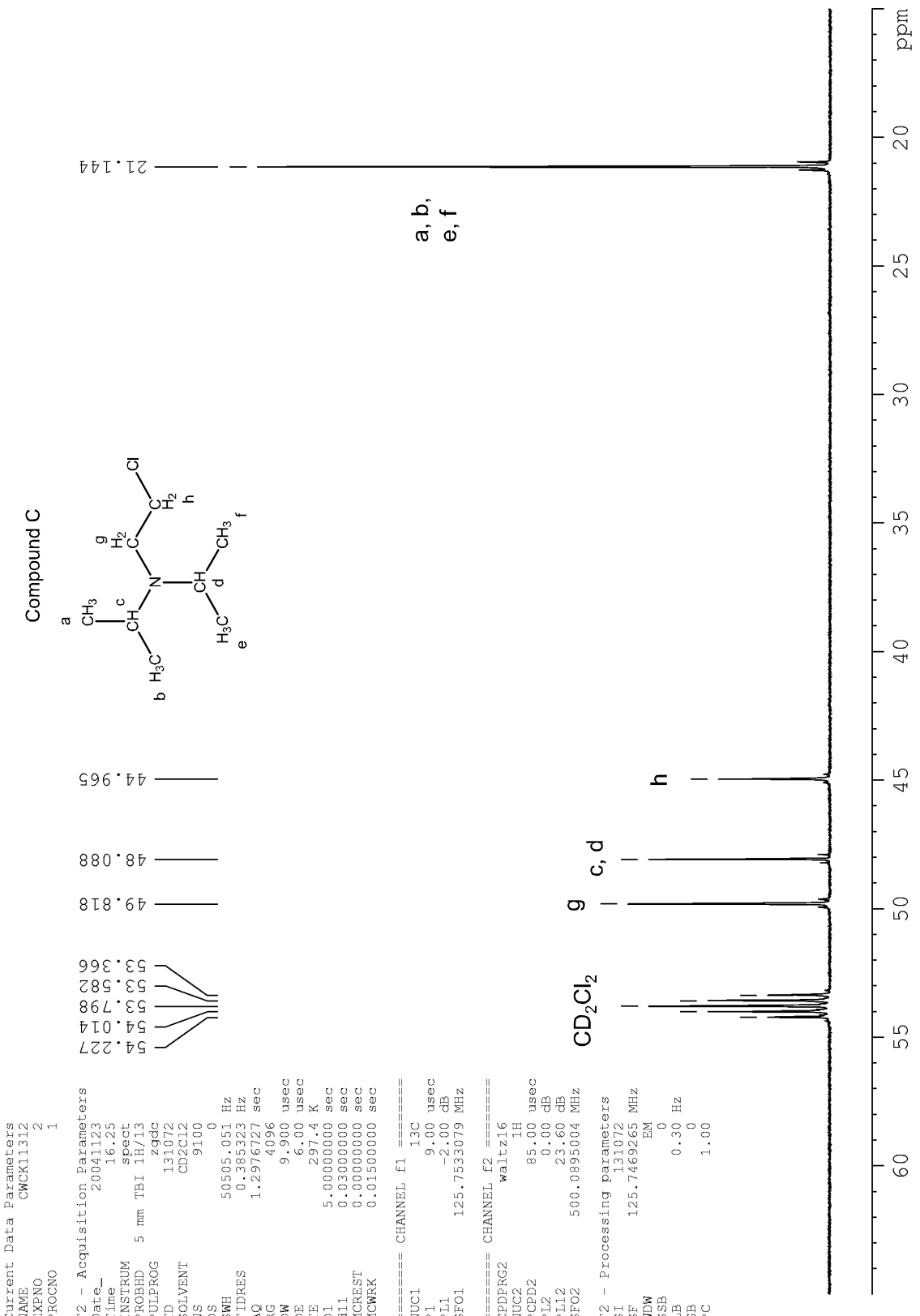
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	125.75 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CH_2Cl_2/CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code) ☐ Other:
☐ Standard addition:	Source :
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.

Experiments:	☐ ^1H ☒ $^{13}C\{^1H\}$ ☐ ^{19}F ☐ $^{31}P\{^1H\}$ ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
a,b,e,f	21.14			
h	44.96			
c,d	48.09			
g	49.82			
Interpretative comments				

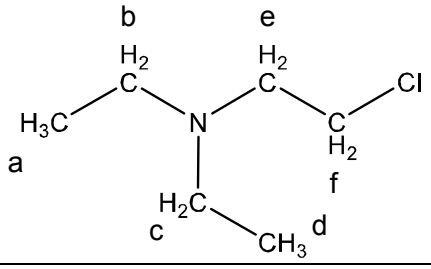


$^{13}C\{^{1}H\}$ spectrum of 2-(N,N-Diisopropylamino)ethylchloride
(Solvent: CD_2Cl_2; Internal reference: CH_2Cl_2 at 53.8 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** D

Aliquot codes: CW-CK-1-88-3	
Sample:	**2-(N,N-Diethylamino)ethylchloride in CH_2Cl_2/CD_2Cl_2**

Compound structure:	

NMR Method name:	**^{1}H NMR**

METHOD DESCRIPTION

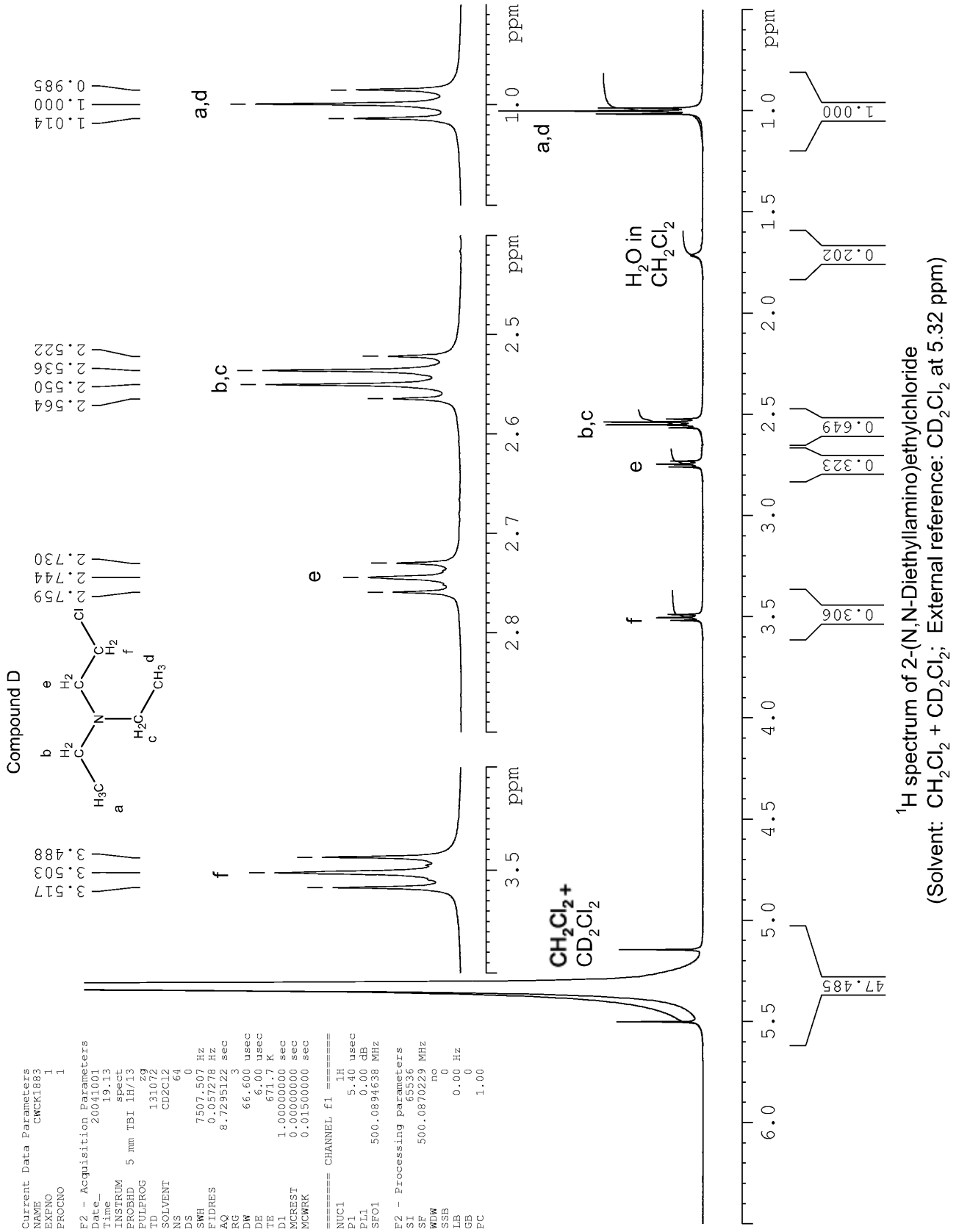
Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	500.09 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CH_2Cl_2/CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source :	☐ Own Synthesis	☐ Commercial
☐ Compared to library spectrum**:**	Source :	☐ OCAD (Code)	☐ Other:
☐ Standard addition:	Source :		
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.		

Experiments: ☒ ^{1}H ☐ ^{13}C{^{1}H} ☐ ^{19}F ☐ ^{31}P{^{1}H} ☐ ^{31}P ☐ COSY ☐ Other:

Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
a,d	1.00		7.0 (b,a; c,d)	
b,c	2.54		7.0 (a,b; d,c)	
e	2.74		7.0 (f,e)	
f	3.50		7.0 (e,f)	
Interpretative comments	Purity = 99.9%			

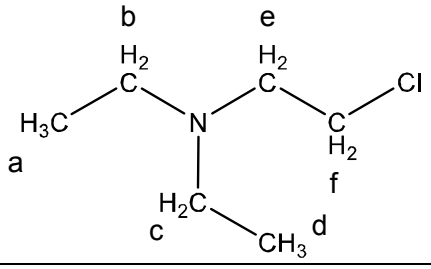


^{1}H spectrum of 2-(N,N-Diethyllamino)ethylchloride
(Solvent: CH_2Cl_2 + CD_2Cl_2; External reference: CD_2Cl_2 at 5.32 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** D

Aliquot codes: CW-CK-1-88-3	
Sample:	**2-(N,N-Diethylamino)ethylchloride in CH_2Cl_2/CD_2Cl_2**

Compound structure:	

NMR Method name:	**$^{13}C\{^1H\}$ NMR**

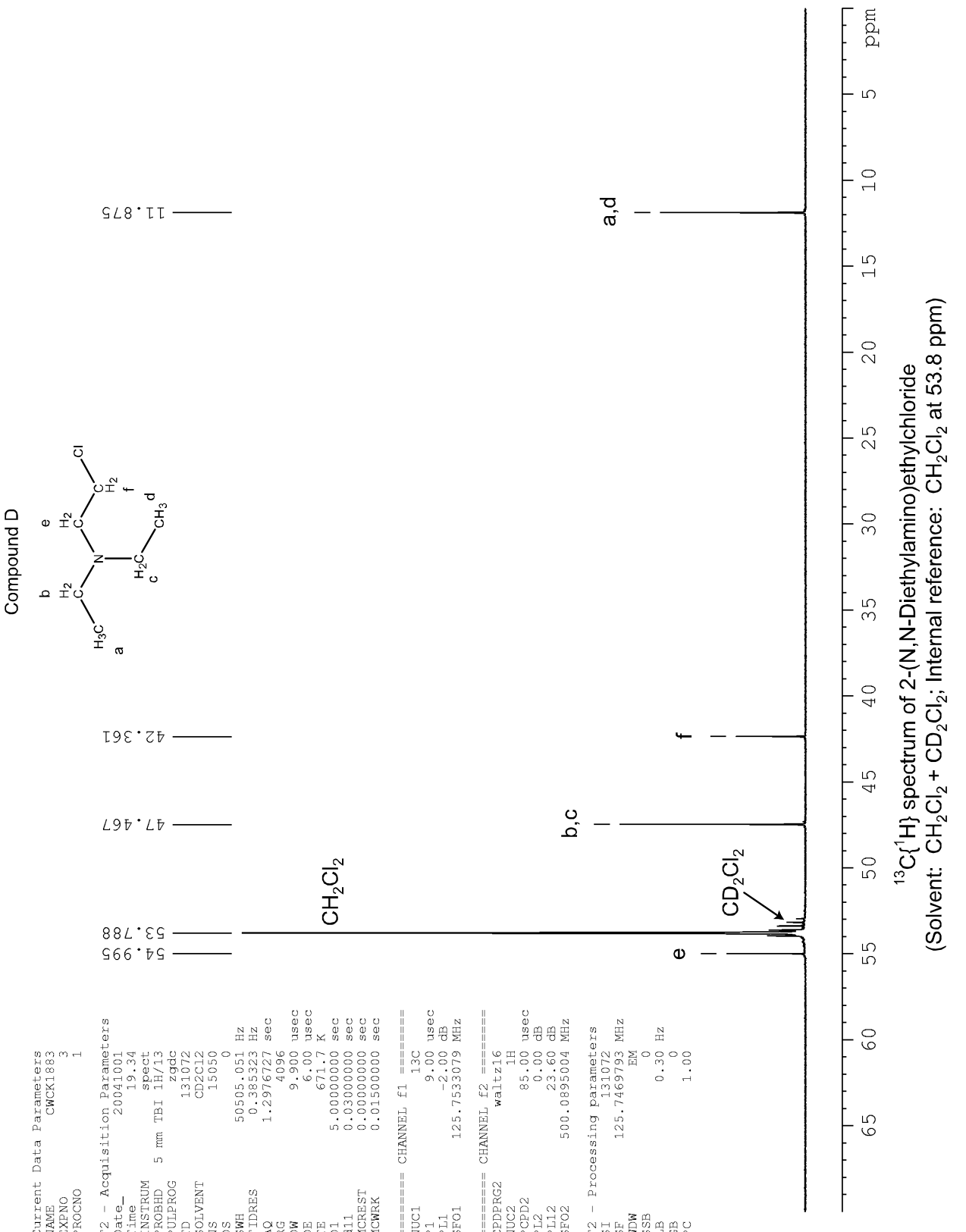
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	125.75 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CH_2Cl_2/CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source :	☐ Own Synthesis	☐ Commercial
☐ Compared to library spectrum**:**	Source :	☐ OCAD (Code)	☐ Other:
☐ Standard addition:	Source :		
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.		

Experiments:	☐ ^1H ☒ $^{13}C\{^1H\}$ ☐ ^{19}F ☐ $^{31}P\{^1H\}$ ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
a,d	11.87			
f	42.36			
b,c	47.47			
e	54.99			
Interpretative comments				

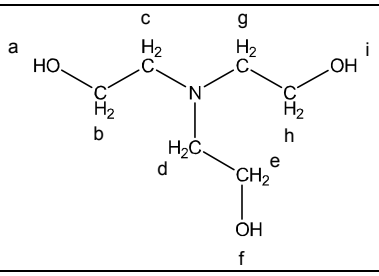


$^{13}C\{^1H\}$ spectrum of 2-(N,N-Diethylamino)ethylchloride
(Solvent: CH_2Cl_2 + CD_2Cl_2; Internal reference: CH_2Cl_2 at 53.8 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** E, J

Aliquot codes: CW-CK-1-90-2	
Sample:	**Triethanolamine in CD_2Cl_2**

Compound structure:	

NMR Method name:	**^{1}H NMR**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker Avance 400		
Frequency:	400.10 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

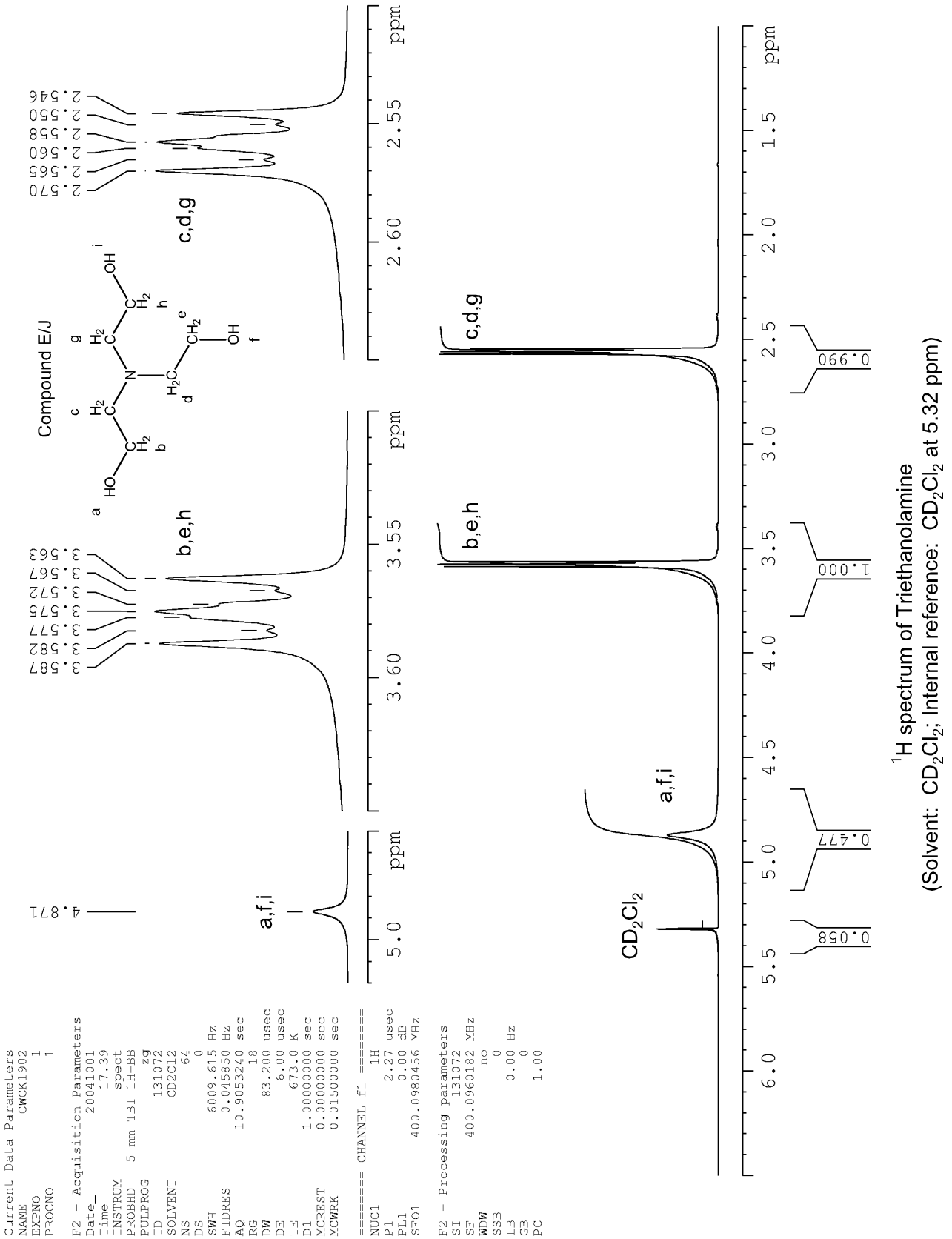
ANALYSIS

☐ Compared to reference chemical**:**	Source :	☐ Own Synthesis	☐ Commercial
☐ Compared to library spectrum**:**	Source :	☐ OCAD (Code)	☐ Other:
☐ Standard addition:	Source :		
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.		

Experiments: ☒ ^{1}H ☐ ^{13}C{^{1}H} ☐ ^{19}F ☐ ^{31}P{^{1}H} ☐ ^{31}P ☐ COSY ☐ Other:

Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
c,d,g	2.56		4.8 (b,c; e,d; h,g)	
b,e,h	3.575		4.8 (c,b; d,e; g,h)	
a,f,i	4.87			

Interpretative comments Purity = 99.7%

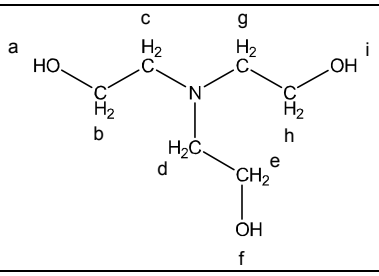


^{1}H spectrum of Triethanolamine
(Solvent: CD_2Cl_2; Internal reference: CD_2Cl_2 at 5.32 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: **Prep Lab** **Sample code(s):** **Purity Check** **Compound number:** **E, J**

Aliquot codes: CW-CK-1-90-2	
Sample:	**Triethanolamine in CD_2Cl_2**

Compound structure:	

NMR Method name:	**$^{13}C\{^1H\}$ NMR**

METHOD DESCRIPTION

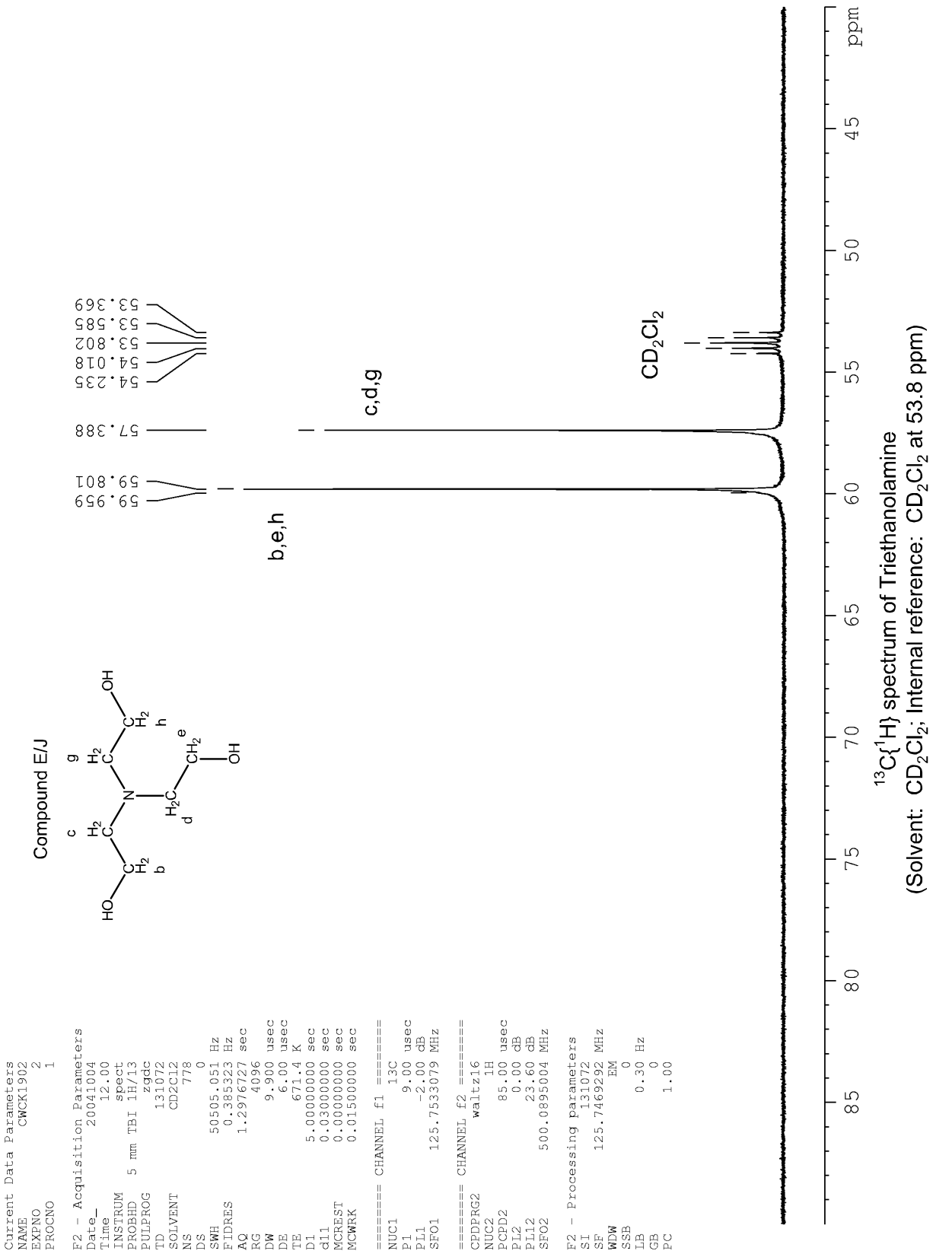
Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	125.75 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CH_2Cl_2/CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis	☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code)	☐ Other:
☐ Standard addition:	Source :	
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.	

Experiments: ☐ ^1H ☒ $^{13}C\{^1H\}$ ☐ ^{19}F ☐ $^{31}P\{^1H\}$ ☐ ^{31}P ☐ COSY ☐ Other:

Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
c,d,g	57.39			
b,e,h	59.80			
Interpretative comments				

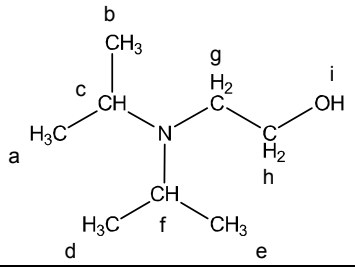


$^{13}C\{^1H\}$ spectrum of Triethanolamine
(Solvent: CD_2Cl_2; Internal reference: CD_2Cl_2 at 53.8 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** G

Aliquot codes: CW-CK-1-90-3	
Sample:	**2-(N,N-Diisopropylamino)ethanol in CD_2Cl_2**

Compound structure:	

NMR Method name:	**^{1}H NMR**

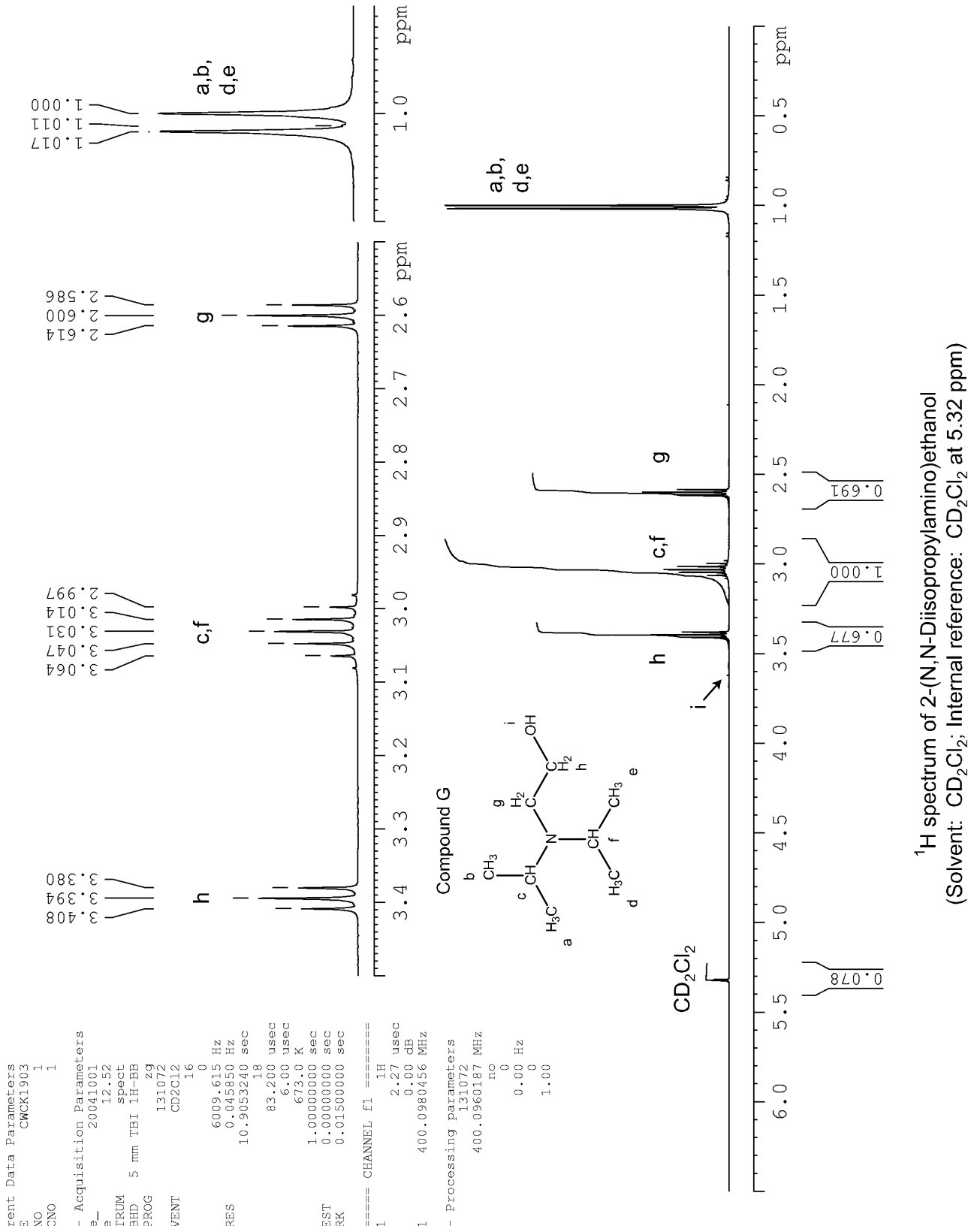
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker Avance 400		
Frequency:	400.10 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code) ☐ Other:
☐ Standard addition:	Source :
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.

Experiments:	☒ ^{1}H ☐ ^{13}C{^{1}H} ☐ ^{19}F ☐ ^{31}P{^{1}H} ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
a,b,d,e	1.01		6.8 (c,a; c,b; f,d; f,e)	
g	2.6		5.6 (h,g)	
c,f	3.03		6.8 (a,c; b,c; d,f; e,f)	
h	3.39		5.6 (g,h)	
Interpretative comments	Purity = 99.8%			

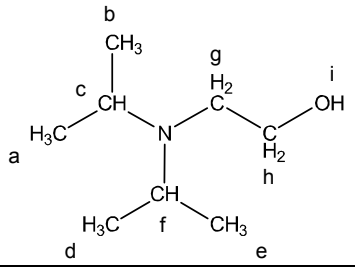


^{1}H spectrum of 2-(N,N-Diisopropylamino)ethanol
(Solvent: CD_2Cl_2; Internal reference: CD_2Cl_2 at 5.32 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** G

Aliquot codes: CW-CK-1-90-3	
Sample:	**2-(N,N-Diisopropylamino)ethanol in CD_2Cl_2**

Compound structure:	

NMR Method name:	**$^{13}C\{^1H\}$ NMR**

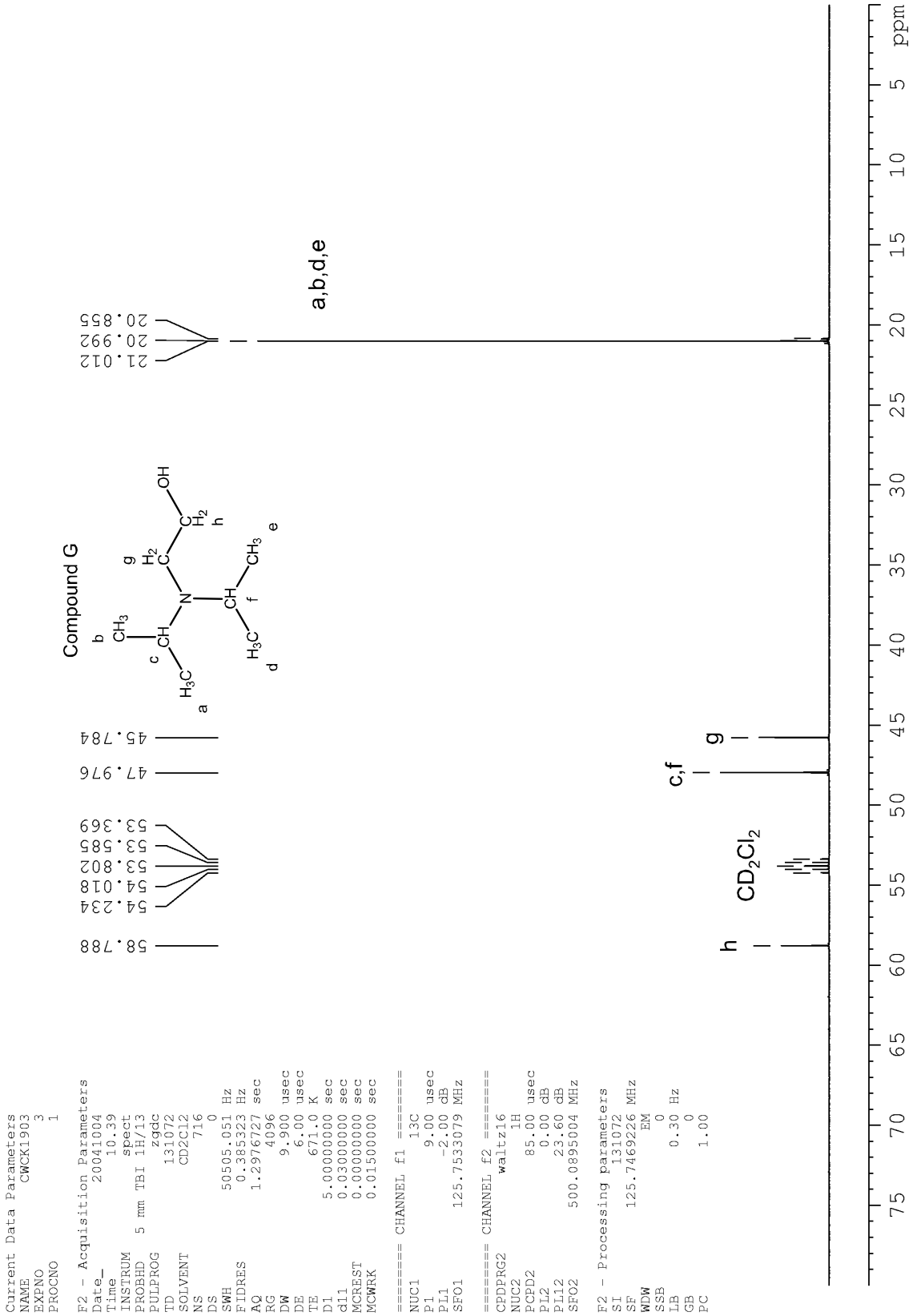
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	125.75 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code) ☐ Other:
☐ Standard addition:	Source :
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.

Experiments:	☐ ^1H ☒ $^{13}C\{^1H\}$ ☐ ^{19}F ☐ $^{31}P\{^1H\}$ ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
a,b,d,e	20.99			
g	45.78			
c,f	47.98			
h	58.79			
Interpretative comments				

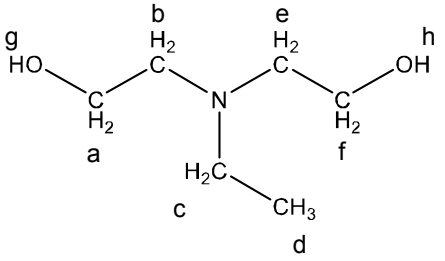


$^{13}C\{^1H\}$ spectrum of 2-(N,N-Diisopropylamino)ethanol
(Solvent: CD_2Cl_2; Internal reference: CD_2Cl_2 at 53.8 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** H

Aliquot codes: CW-CK-1-91-2	
Sample:	**Ethyldiethanolamine in CD_2Cl_2**

Compound structure:	

NMR Method name:	**^{1}H NMR**

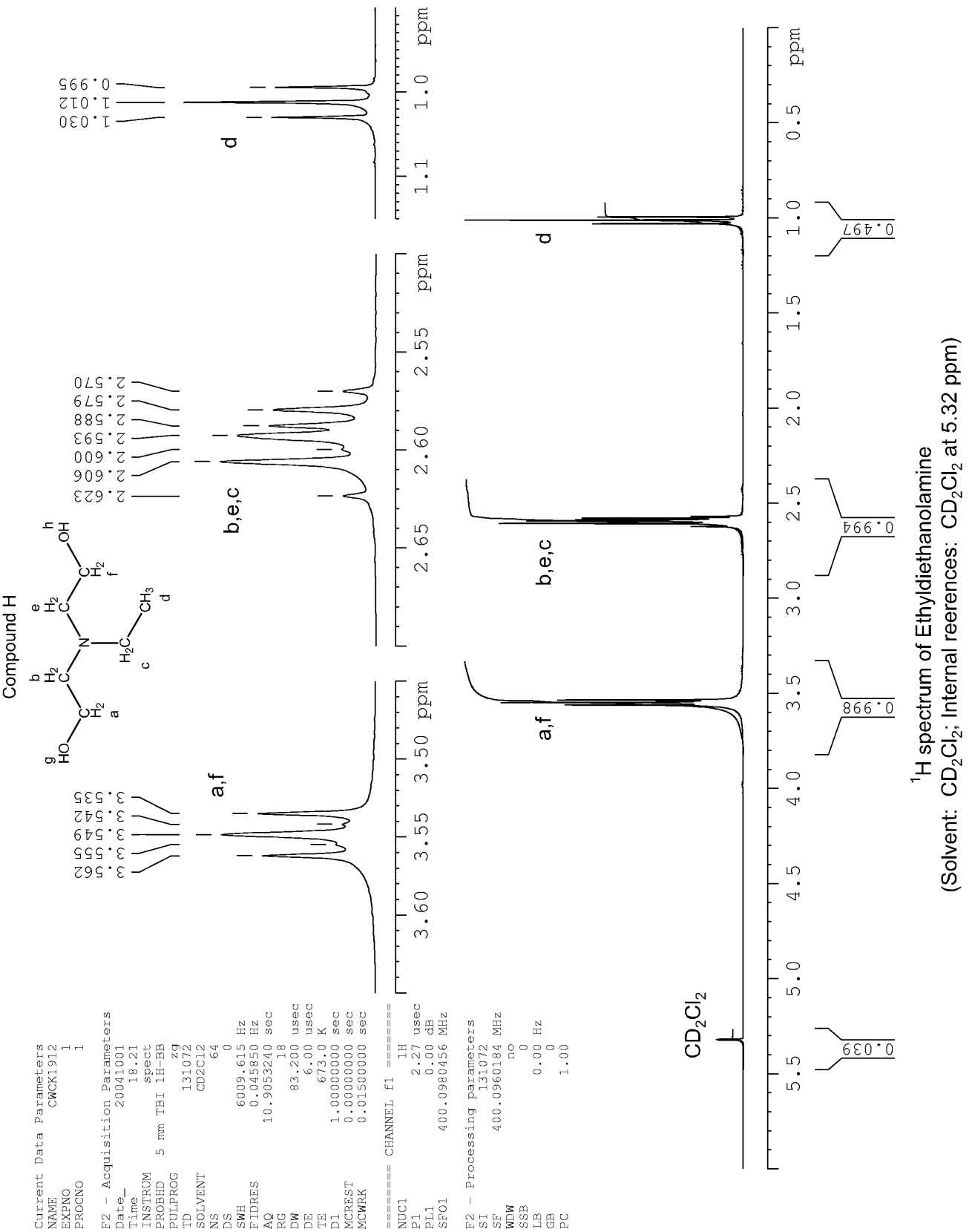
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker Avance 400		
Frequency:	400.10 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code) ☐ Other:
☐ Standard addition:	Source :
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.

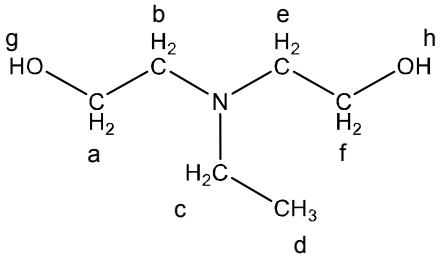
Experiments:	☒ ^{1}H ☐ ^{13}C{^{1}H} ☐ ^{19}F ☐ ^{31}P{^{1}H} ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
d	1.01		7.2 (c,d)	
b,e,c	2.59			
a,f	3.55		5.6 (b,a; e,f)	
Interpretative comments	Purity = 99.6%			


Compound H
Current Data Parameters
NAME CWCK1912
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041001
Time 18.21
INSTRUM spect
PROBHD 5 mm TBI 1H-BB
PULPROG zg
TD 131072
SOLVENT CD2Cl2
NS 64
DS 0
SWH 6009.615 Hz
FIDRES 0.045850 Hz
AQ 10.9053240 sec
RG 18
DW 83.200 usec
DE 6.00 usec
TE 673.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 2.27 usec
PL1 0.00 dB
SFO1 400.0980456 MHz
F2 - Processing parameters
SI 131072
SF 400.0960184 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
a,f
b,e,c
d
CD2Cl2
0.039
0.998
0.994
0.497
ppm
1H spectrum of Ethyldiethanolamine
(Solvent: CD2Cl2; Internal reerences: CD2Cl2 at 5.32 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** H

Aliquot codes: CW-CK-1-91-2	
Sample:	**Ethyldiethanolamine in CD_2Cl_2**

Compound structure:	

NMR Method name:	**$^{13}C\{^1H\}$ NMR**

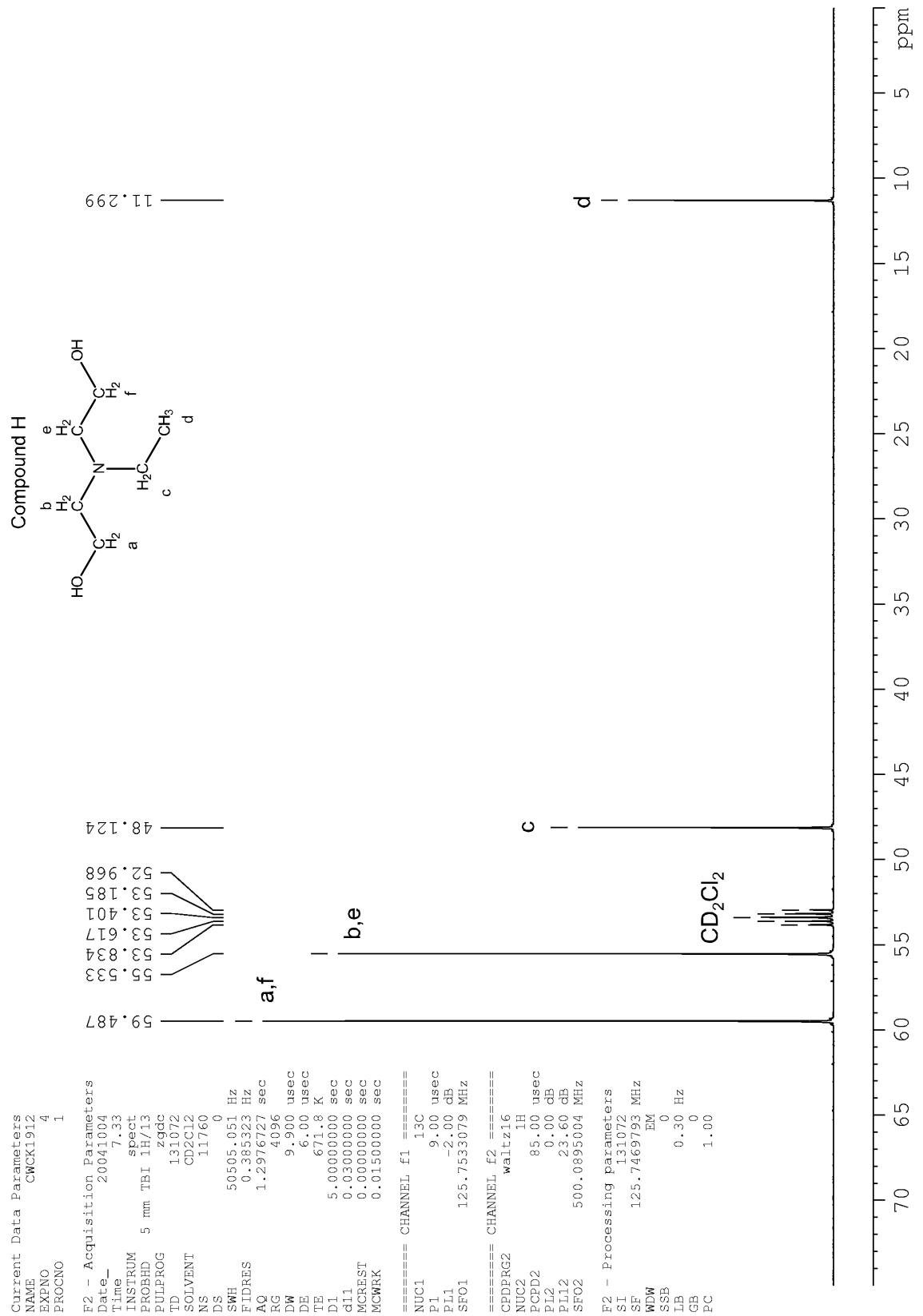
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	125.75 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code) ☐ Other:
☐ Standard addition:	Source :
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.

Experiments:	☐ ^1H ☒ $^{13}C\{^1H\}$ ☐ ^{19}F ☐ $^{31}P\{^1H\}$ ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
d	11.30			
c	48.12			
b,e	55.53			
a,f	59.49			
Interpretative comments				

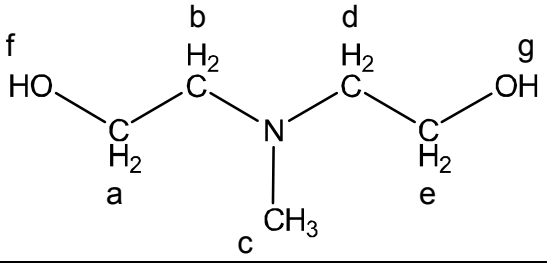


$^{13}C\{^{1}H\}$ spectrum of Ethyldiethanolamine
(Solvent: CD_2Cl_2; Internal reference: CD_2Cl_2 at 53.8 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** I

Aliquot codes: CW-CK-1-91-1	
Sample:	**Methyldiethanolamine in CD_2Cl_2**

Compound structure:	

NMR Method name:	**^{1}H NMR**

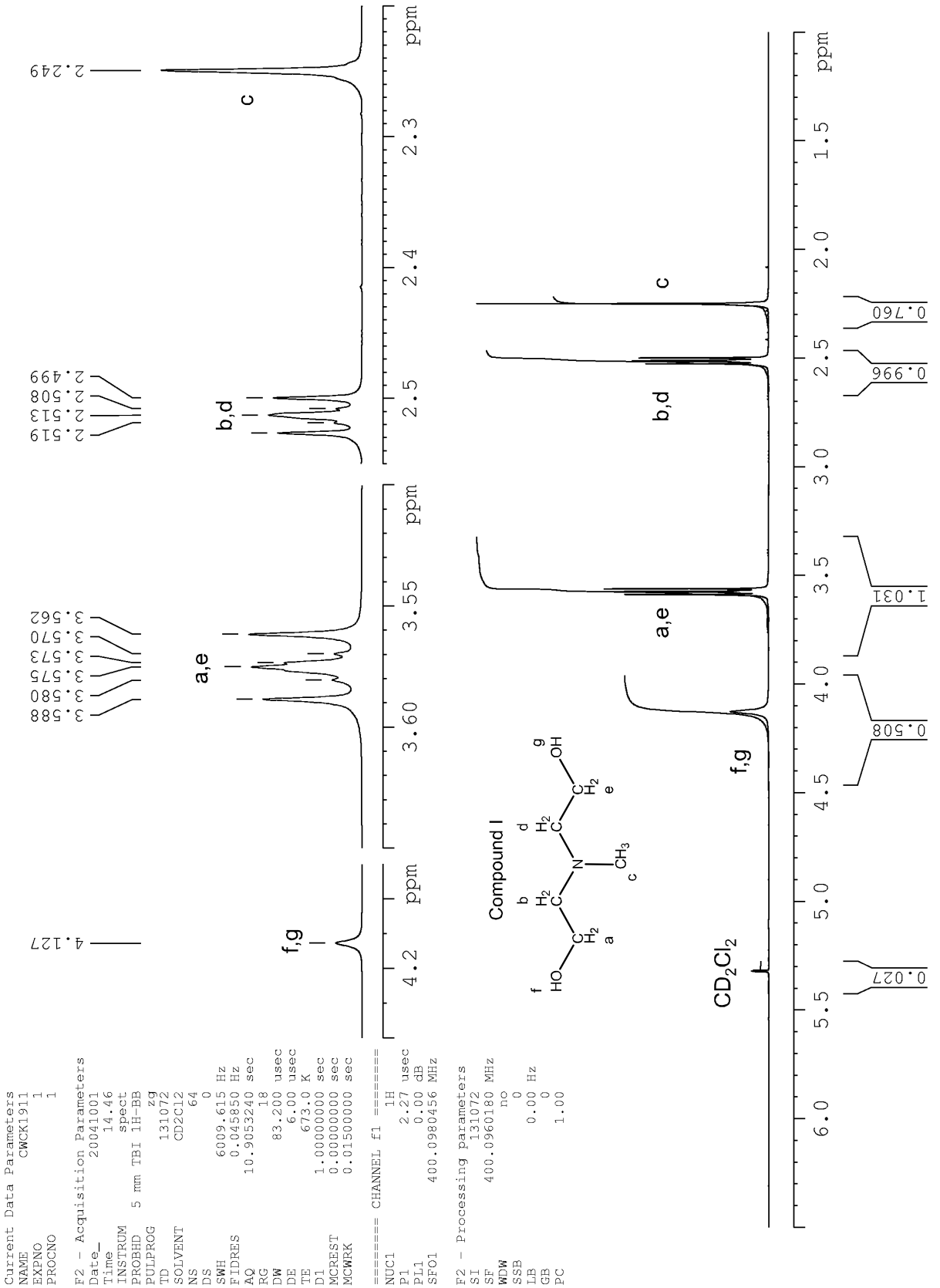
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker Avance 400		
Frequency:	400.10 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code) ☐ Other:
☐ Standard addition:	Source :
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.

Experiments:	☒ ^{1}H ☐ ^{13}C{^{1}H} ☐ ^{19}F ☐ ^{31}P{^{1}H} ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
c	2.25			
b,d	2.51		5.6 (a,b; d,e)	
a,e	3.57		5.6 (b,a; e,d)	
f,g	4.13			
Interpretative comments	Purity = 99.7%			

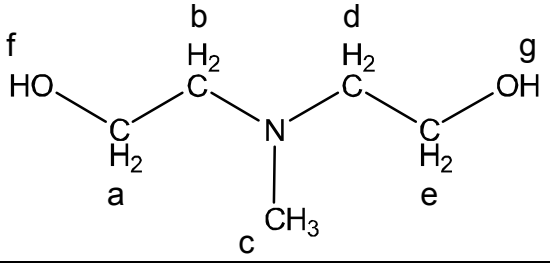


^{1}H spectrum of Methyldiethanolamine
(Solvent: CD_2Cl_2; Internal reference: CD_2Cl_2 at 5.32 ppm)

NMR METHOD AND ANALYSIS RESULTS

Laboratory code: Prep Lab **Sample code(s):** Purity Check **Compound number:** I

Aliquot codes: CW-CK-1-91-1	
Sample:	**Methyldiethanolamine in CD_2Cl_2**

Compound structure:	

NMR Method name:	**$^{13}C\{^1H\}$ NMR**

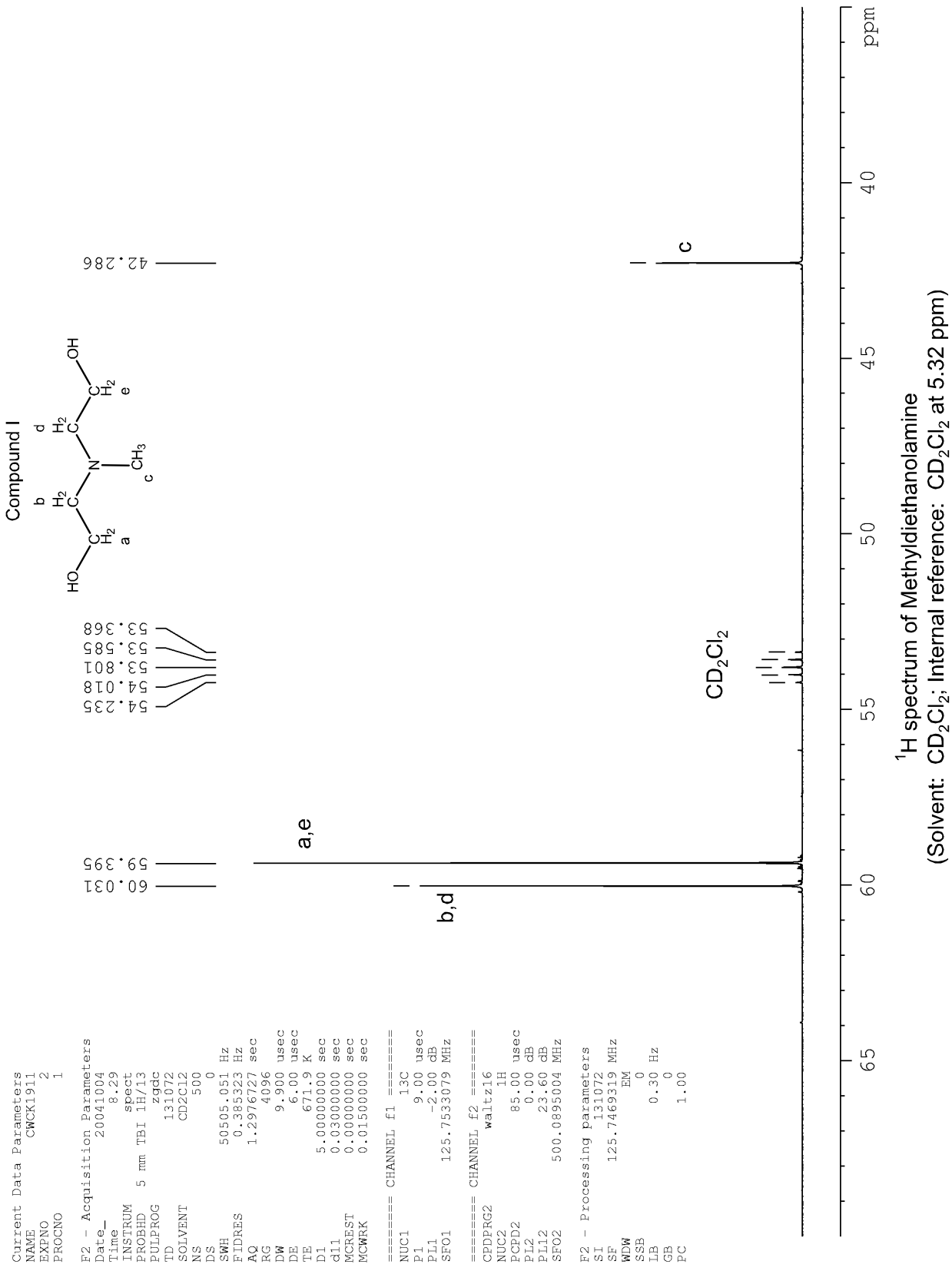
METHOD DESCRIPTION

Instrument Manufacturer and Type:	Bruker DRX 500		
Frequency:	125.75 MHz	**Temperature control unit:**	☒ Yes ☐ No
Probe head:	TBI	**Temperature:**	26.5 °C
Sample tube diameter:	5 mm	**Solvent:**	**CD_2Cl_2**
pH of:	**Sample** =	**Blank** =	**Reference** =
δ reference reagent in Sample:	☒ Internal = **CD_2Cl_2**	☐ External =	
δ reference reagent in Reference:	☐ Internal =	☐ External =	

ANALYSIS

☐ Compared to reference chemical**:**	Source : ☐ Own Synthesis	☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (Code)	☐ Other:
☐ Standard addition:	Source :	
☒ Spectral interpretation:	E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.	

Experiments:	☐ ^1H ☒ $^{13}C\{^1H\}$ ☐ ^{19}F ☐ $^{31}P\{^1H\}$ ☐ ^{31}P ☐ COSY ☐ Other:			
Assignment(s):	**Chemical shift(s)**		**Coupling constant(s)**	
	Sample spectrum [ppm]	**Reference spectrum [ppm]**	**Sample spectrum [Hz]**	**Reference spectrum [Hz]**
c	42.29			
a,e	59.39			
b,d	60.03			
Interpretative comments				


Current Data Parameters
NAME CWCK1911
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041004
Time 8.29
INSTRUM spect
PROBHD 5 mm TBI 1H/13
PULPROG zgdc
TD 131072
SOLVENT CD2Cl2
NS 500
DS 0
SWH 50505.051 Hz
FIDRES 0.385323 Hz
AQ 1.2976727 sec
RG 4096
DW 9.900 usec
DE 6.00 usec
TE 671.9 K
D1 5.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 -2.00 dB
SFO1 125.7533079 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 0.00 dB
PL12 23.60 dB
SFO2 500.0895004 MHz
F2 - Processing parameters
SI 131072
SF 125.7469319 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Compound I
HO
OH
N
CH3
a
b
c
d
e
60.031
59.395
a,e
b,d
54.235
53.018
52.801
52.585
52.368
42.286
CD2Cl2
c
65
60
55
50
45
40
ppm
1H spectrum of Methyldiethanolamine
(Solvent: CD2Cl2; Internal reference: CD2Cl2 at 5.32 ppm)

ORGANISATION FOR THE PROHIBITION OF CHEMICAL WEAPONS

Report of the Sixteenth Official OPCW Proficiency Test

Part III: Qualitative Analysis

Laboratory code: Sample Prep Lab

TABLE OF CONTENTS

SUMMARY: PARTICIPATING LABORATORY

1. Participating laboratory

Laboratory code	Sample Prep Lab, aliquot #31
Name of the laboratory/institute participating in the test	Lawrence Livermore National Laboratory
Contact person	Mr. Armando Alcaraz
Address	PO Box 808, M/S L-178 7000 East Avenue Livermore, CA 94551
Telephone number	925-423-6889
Fax number	925-423-9014
Email address	Alcaraz1@llnl.gov

2. Analysts and authentication

	Name	Title	Pages*	Date**	Signature**
1	Mr. Armando Alcaraz	Principal Investigator	All	Nov. 30, 2004	
2	Dr. Hugh Gregg	Co-PI, Senior Chemist	All	Nov. 30, 2004	
3	Dr. Carolyn Koester	Research Scientist	Prep & GC/MS	Nov. 30, 2004	
4	Dr. Phil Pagoria	Research Scientist	Synthesis	Nov. 30, 2004	
5	Dr. Andrew Vance	Research Scientist	Synthesis	Nov. 30, 2004	
6	Dr. Robert Maxwell	Research Scientist	NMR analysis	Nov. 30, 2004	
7	Dr. Sarah Chinn	Research Scientist	NMR analysis	Nov. 30, 2004	
8	Mr. Rich Whipple	Scientist	Sample Prep	Nov. 30, 2004	
9	Ms. Tuijauna Mitchell-Hall	QA Manager	QA/QC	Nov. 30, 2004	
10					
11					
12					

* Page numbers defining the responsibility area of the analyst;
** Date and signature of the responsible analyst;

SUMMARY: QUALITY ASSURANCE / QUALITY CONTROL (QA/QC)

1. Status of the laboratory (tick where applicable)

☒ Accreditation accepted:
Year 2001 Accreditation body: American Association for Laboratory Accreditation
Scope of accreditation: Chemical

☐ Accreditation planned/pending:
Target year _____ Accreditation body: _____
Scope of accreditation: _____

☐ Not accredited.

2. Quality system (tick where applicable)

☒ Described in a Quality Assurance Manual/Handbook. Quality system in accordance with:
☐ ISO 900_____, ☐ EN 4500_____, ☒ ISO Guide 17025, ☐ Other: _____

☐ No quality system. Please, fill in question number 3.

3. QA/QC Summary (Summary of the applied quality assurance and quality control (QA/QC) procedures concerning sample preparation, calibration, and analysis. Requested only from laboratories without a quality system).

THE AMERICAN
ASSOCIATION
FOR LABORATORY
ACCREDITATION

ACCREDITED LABORATORY

A2LA has accredited

LAWRENCE LIVERMORE NATIONAL LABORATORY
Livermore, CA

for technical competence in the field of

Chemical Testing

The accreditation covers the specific tests and types of tests listed on the agreed scope of accreditation. This laboratory meets the requirements of ISO/IEC 17025 - 1999 "General Requirements for the Competence of Testing and Calibration Laboratories" and any additional program requirements in the identified field of testing.

Presented this 12th day of April 2004.

President
For the Accreditation Council
Certificate Number 1914-01
Valid to February 28, 2006

For tests or types of tests to which this accreditation applies, please refer to the laboratory's Chemical Scope of Accreditation.

American Association for Laboratory Accreditation

SCOPE OF ACCREDITATION TO ISO/IEC 17025-1999

LAWRENCE LIVERMORE NATIONAL LABORATORY
FORENSIC SCIENCE CENTER – OPCW PROJECT
7000 East Avenue Mailstop L-178
Livermore, CA 94550
Armando Alcaraz Phone: 925 423 6889

CHEMICAL

Valid To: February 28, 2006 Certificate Number: 1914-01

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform the following types of Qualitative Tests for Chemicals related to Chemical Warfare Convention (CWC) in unknown samples:

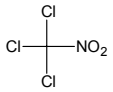
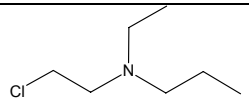
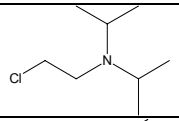
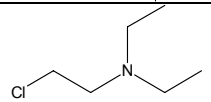
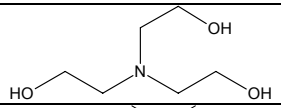
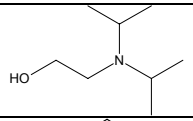
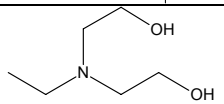
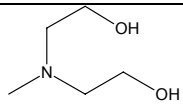
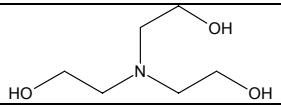
Sample Preparation	BB-SP5, BB-SP6, BB-SP8, BB-SP9, LL-SP1, LL-SP2, LL-SP3, LL-SP4
Test	**Procedures**
Spectroscopy	
Nuclear magnetic resonance	BB-NMR1
Capillary zone electrophoresis / UV detection	LL-CE1
Gas chromatography / Fourier Transform Infrared Spectrometry	BB-IR1
Chromatography	
Gas chromatography / Element Specific Detectors	BB-GC1
Gas chromatography / Mass Spectrometry	BB-MS1
Liquid chromatography / Atmospheric pressure Chemical Ionization / Mass Spectrometry	BB-MS4
Liquid Chromatography / Electrospray Ionization Mass Spectrometry	LL-MS1
Chain of Custody for Laboratory	LL-CC1
Work Instructions for the Preparation of Test Samples for OPCW Proficiency Tests	QDOC/LAB/WI/ PT2 (Minus Section 11 Confirmation)

(A2LA Cert. No. 1914-01) Revised 04/15/2004

5301 Buckeystown Pike, Suite 350 • Frederick, MD 21704-8373 • Phone: 301-644 3248 • Fax: 301-662 2974

SUMMARY: NAMES AND STRUCTURES OF ALL REPORTED COMPOUNDS

Laboratory code: 31

Sam. code	Cmpd. no*	Compound name	Chemical Abstract number	Compound Structure	Molecular formula	Schedule number	Comments**
O	1	Trichloronitromethane	76-06-2		CCl_3NO_2	3.A.04	
O	2	2-(N-Ethyl-N-propylamino)ethylchloride			$C_7H_{16}ClN$	2.B.10	
O	3	2-(N,N-Diisopropylamino)ethylchloride	96-79-7		$C_8H_{18}ClN$	2.B.10	
O	4	2-(N,N-Diethylamino)ethylchloride	100-35-6		$C_6H_{14}ClN$	2.B.10	
L	5	Triethanolamine	102-71-6		$C_6H_{15}NO_3$	3.B.17	
L	6	2-(N,N-Diisopropylamino)ethanol	96-80-0		$C_8H_{19}NO$	2.B.11	
S	7	Ethyldiethanolamine	139-87-7		$C_6H_{15}NO_2$	3.B.15	
S	8	Methyldiethanolamine	105-59-9		$C_5H_{13}NO_2$	3.B.16	
S	9	Triethanolamine	102-71-6		$C_6H_{15}NO_3$	3.B.17	

* Compound number defined by the participating laboratory and used throughout the report for the reported compound.

** Explanation for the reporting of non-scheduled compounds, details can be added in the comment section of the report.

Note: There must be an unbroken chain of evidence linking each reported chemical to the original sample from which an aliquot was prepared and analyzed for the identification of this chemical.

SUMMARY: ANALYTICAL TECHNIQUES

Laboratory code: 31 Sample code(s): O/31

Compound number*	Compound name	Compound analysed as	Analytical technique	Method name	Method page no.	Aliquot name
1	**Trichloronitromethane**	☒ original compound ☐ methylated ☐ silylated ☐ other: _____	GC/MS-EI GC/MS-CI	CW CW-CI	70 73	CW-1-130-7-O CW-1-130-7-O
2	**2-(N-Ethyl-N-propylamino)ethylchloride**	☒ original compound ☐ methylated ☐ silylated ☐ other: _____	GC/MS-EI GC/MS-CI	CW CW-CI	76 79	CW-1-130-7-O CW-1-130-7-O
3	**2-(N,N-Diisopropylamino)ethylchloride**	☒ original compound ☐ methylated ☐ silylated ☐ other: _____	GC/MS-EI GC/MS-CI	CW CW-CI	82 85	CW-1-130-7-O CW-1-130-7-O
4	**2-(N,N-Diethylamino)ethylchloride**	☒ original compound ☐ methylated ☐ silylated ☐ other: _____	GC/MS-EI GC/MS-CI	CW CW-CI	88 91	CW-1-130-7-O CW-1-130-7-O

* Compound number defined by the participating laboratory (see Summary: Names and Structures of All Reported Compounds);

SUMMARY: ANALYTICAL TECHNIQUES

Laboratory code: 31 Sample code(s): L/31

Compound number*	Compound name	Compound analysed as	Analytical technique	Method name	Method page no.	Aliquot name
5	**Triethanolamine**	☐ original compound ☐ methylated ☒ silylated ☐ other: _____	GC/MS-EI GC/MS-CI	TMS_A CW-CI-TM	96 99	CW-1-131-5-L CW-1-131-5-L
6	**2-(N,N-Diisopropylamino)ethanol**	☐ original compound ☐ methylated ☒ silylated ☐ other: _____	GC/MS-EI GC/MS-CI	TMS_A CW-CI-TM	102 105	CW-1-131-4-L CW-1-131-4-L
		☐ original compound ☐ methylated ☐ silylated ☐ other: _____				
		☐ original compound ☐ methylated ☐ silylated ☐ other: _____				

* Compound number defined by the participating laboratory (see Summary: Names and Structures of All Reported Compounds);

SUMMARY: ANALYTICAL TECHNIQUES

Laboratory code: 31 Sample code(s): S/31

Compound number*	Compound name	Compound analysed as	Analytical technique	Method name	Method page no.	Aliquot name
7	**Ethyldiethanolamine**	☐ original compound ☐ methylated ☒ silylated ☐ other: _____	GC/MS-EI GC/MS-CI	TMS_A CW-CI-TM	110 113	CW-1-131-2-S CW-1-131-2-S
8	**Methyldiethanolamine**	☐ original compound ☐ methylated ☒ silylated ☐ other: _____	GC/MS-EI GC/MS-CI	TMS_A CW-CI-TM	116 119	CW-1-131-2-S CW-1-131-2-S
9	**Triethanolamine**	☐ original compound ☐ methylated ☒ silylated ☐ other: _____	GC/MS-EI GC/MS-CI	TMS_A CW-CI-TM	122 125	CW-1-131-2-S CW-1-131-2-S
		☐ original compound ☐ methylated ☐ silylated ☐ other: _____				

* Compound number defined by the participating laboratory (see Summary: Names and Structures of All Reported Compounds);

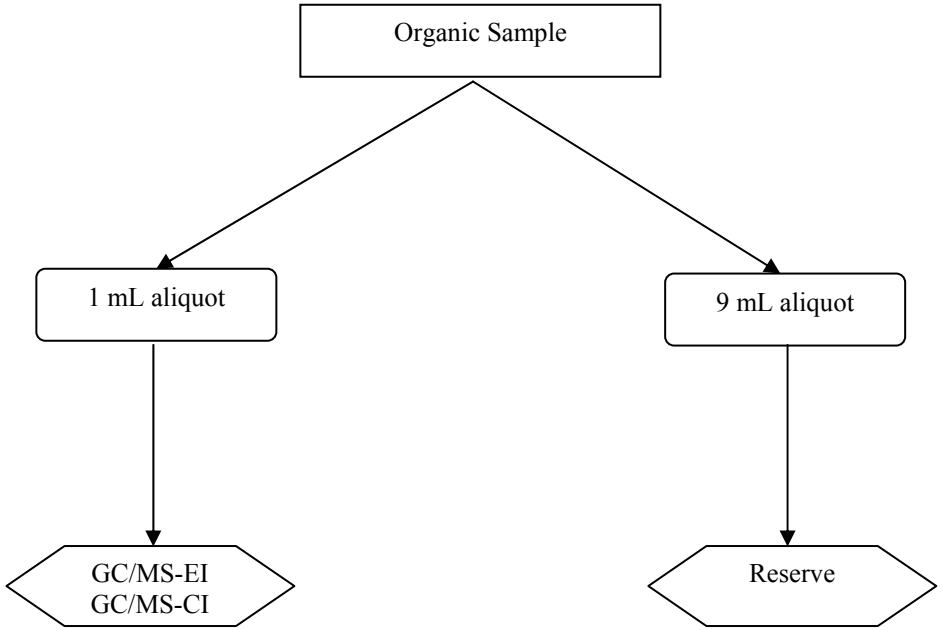
SAMPLE PREPARATION DESCRIPTION

Laboratory code: 31 Sample code(s): O/31 Sample Blank code: OB/31

1. Sample preparation

Sample/ Aliquot Code	*Specification of Sample/ Type of Sample Preparation*	**Amount/ Volume**	*Sample Preparation Procedures*	**End Volume**	**Resulting Aliquot Code**	**Analytical Technique(s)**
O/31 OB/31	1 mL aliquot of each	1 mL	(none)	1 mL	CW-1-130-7-O CW-1-130-8-OB	GC/MS-EI GC/MS-CI

2. Additional information



Note: This flowchart is for visualization only; see the preceding sample preparation description page for sample aliquot numbers

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** O/31, OB/31 **Compound number:** 1

Aliquot codes:			
Sample:	**CW-1-130-7-O**	**Blank:**	**CW-1-130-8-OB**

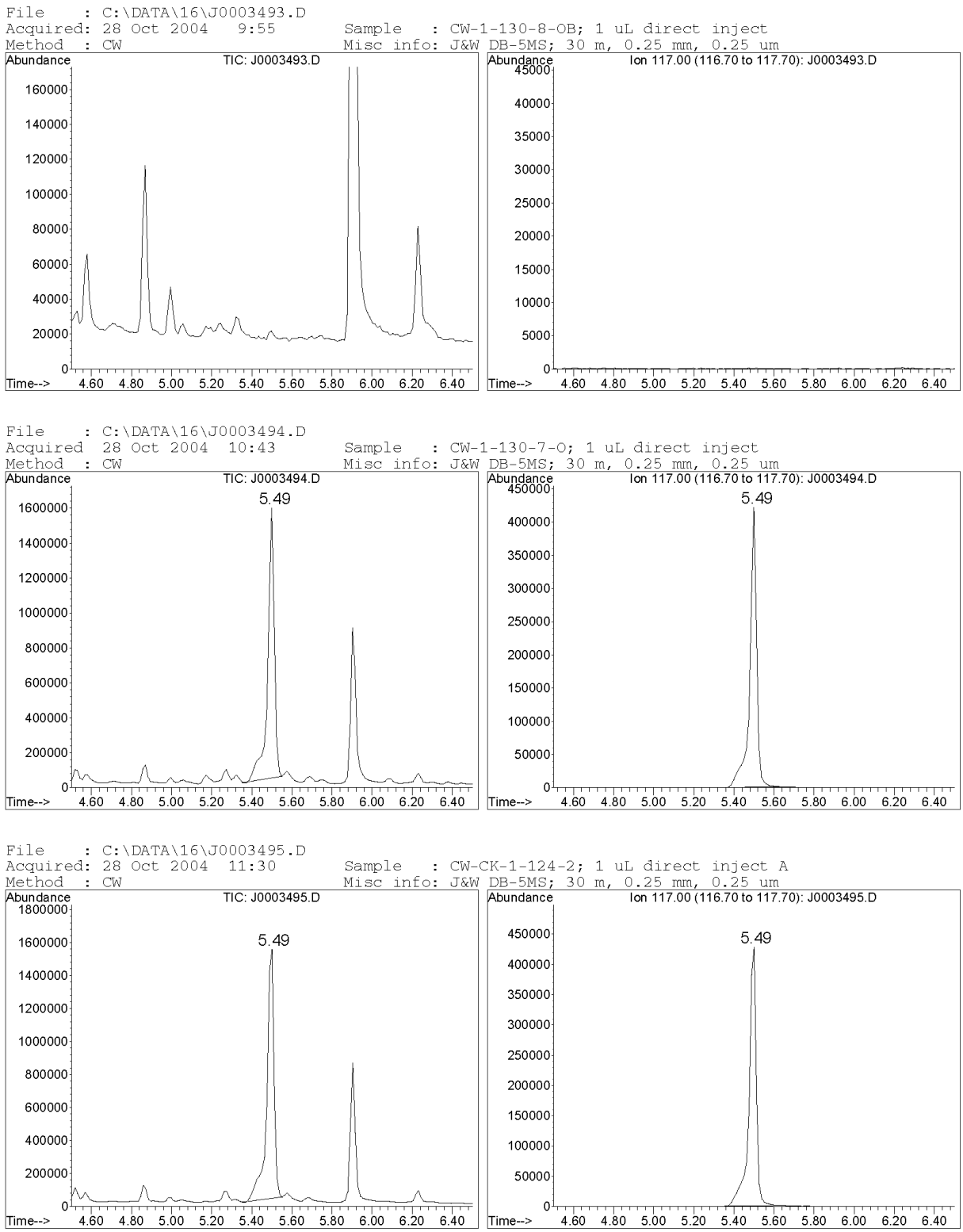
GC-EI-MS Method name:	**CW**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	3 min	**Scan range:**	30-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☒ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



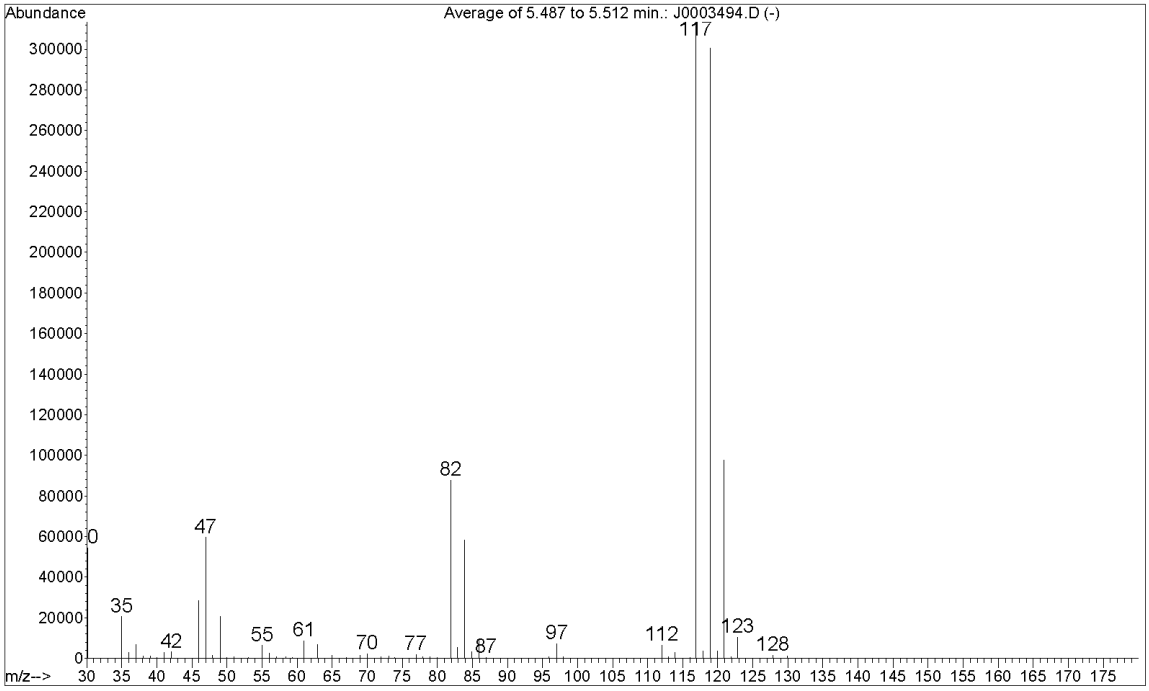
EI chromatograms supporting identification of compound **1**; TIC on left; EIC (m/z **117**) on right.

Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

Center: Chromatograms of Organic sample, aliquot **CW-1-130-7-O** from **O/31**, retention time **5.49** min.

Bottom: Chromatograms of authentic reference standard of **Trichloronitromethane**, retention time **5.49** min.

```
File       : C:\DATA\16\J0003494.D
Acquired   : 28 Oct 2004  10:43     using AcqMethod CW
Sample Name: CW-1-130-7-O; 1 uL direct inject
Misc Info  : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um
```




```
File       : C:\DATA\16\J0003495.D
Acquired   : 28 Oct 2004  11:30     using AcqMethod CW
Sample Name: CW-CK-1-124-2; 1 uL direct inject A
Misc Info  : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um
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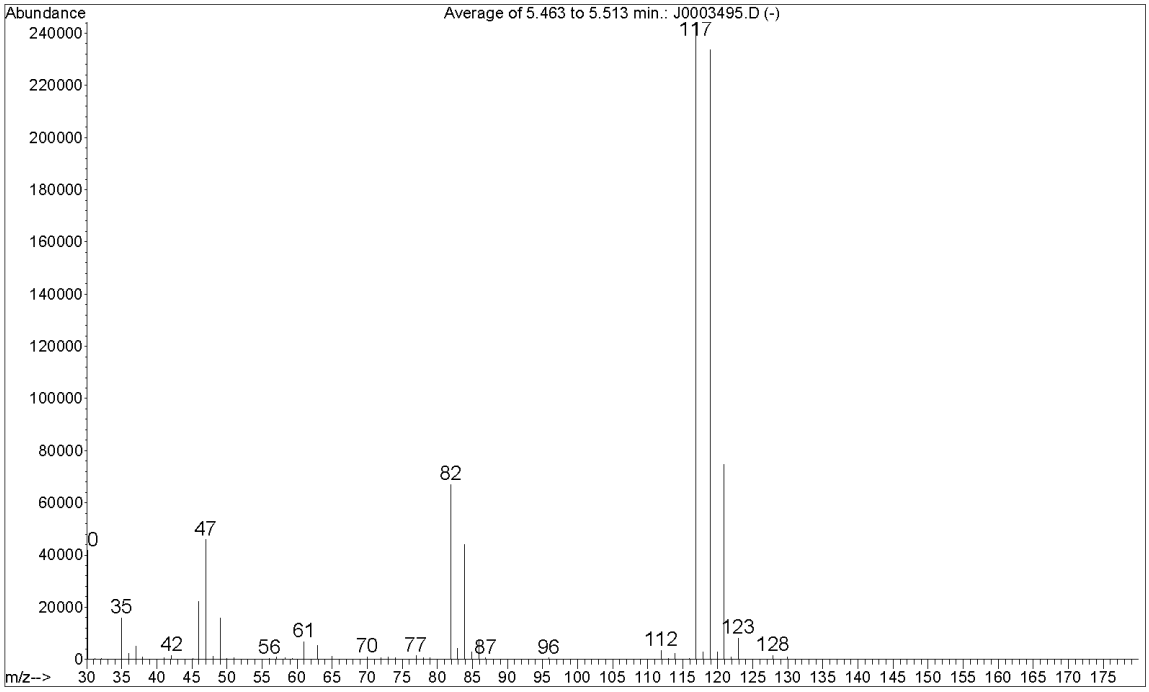


EI mass spectrum of:

Top: Compound **1** in Organic sample **O/31**, aliquot **CW-1-130-7-O**

Bottom: Authentic reference standard of **Trichloronitromethane** corresponding to compound **1** (MW: **163**)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** O/31, OB/31 **Compound number:** 1

Aliquot codes:			
Sample:	**CW-1-130-7-O**	**Blank:**	**CW-1-130-8-OB**

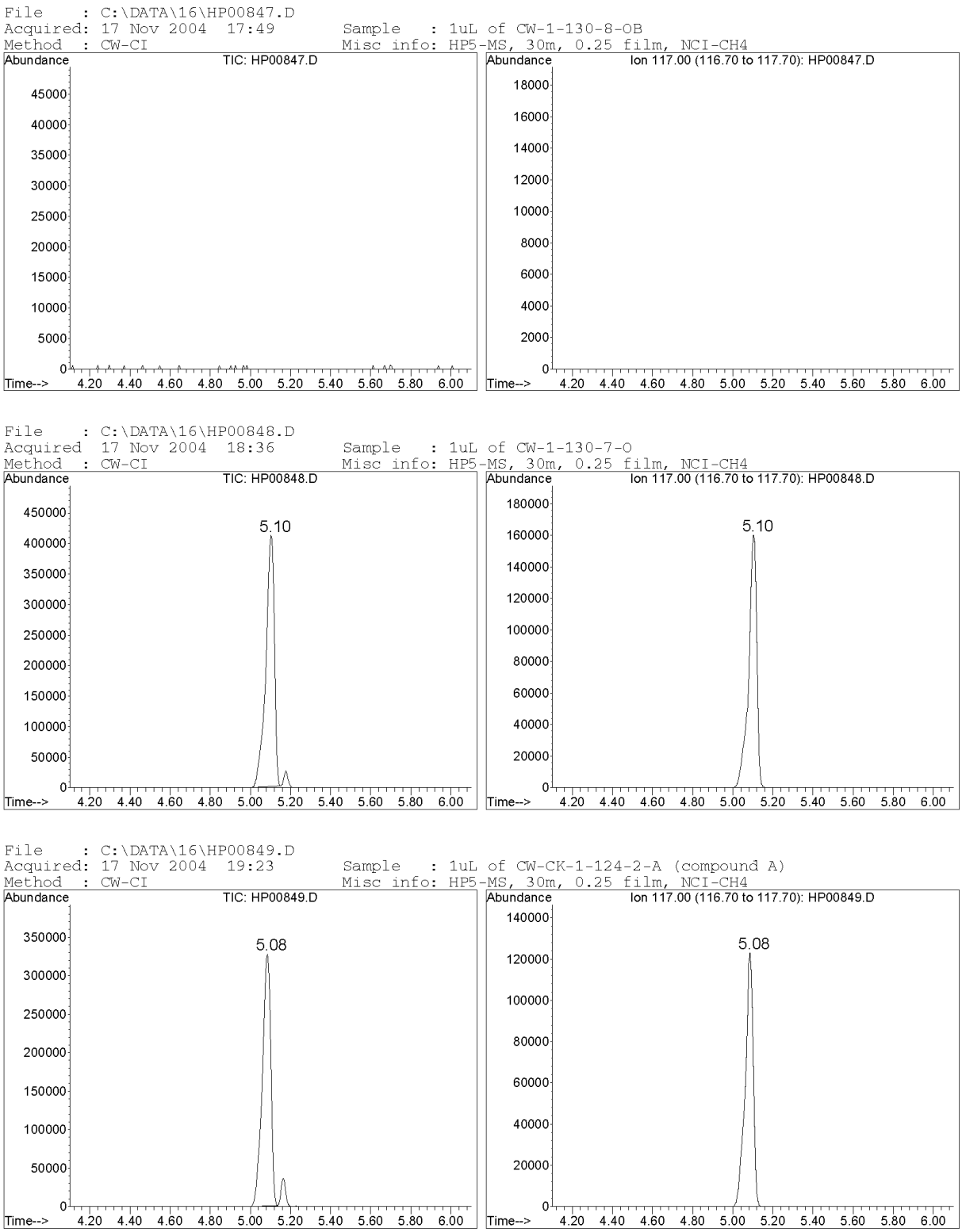
GC-CI-MS Method name:	**CW-CI**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☒ Methane ☐ Isobutane ☐ Ammonia ☐ Other:		
Solvent delay time:	3 min	**Scan range:**	50-550 m/z
Electron energy:	235 eV	**Scan time:**	0.35 s
Ionisation polarity:	☐ Positive ☒ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☒ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



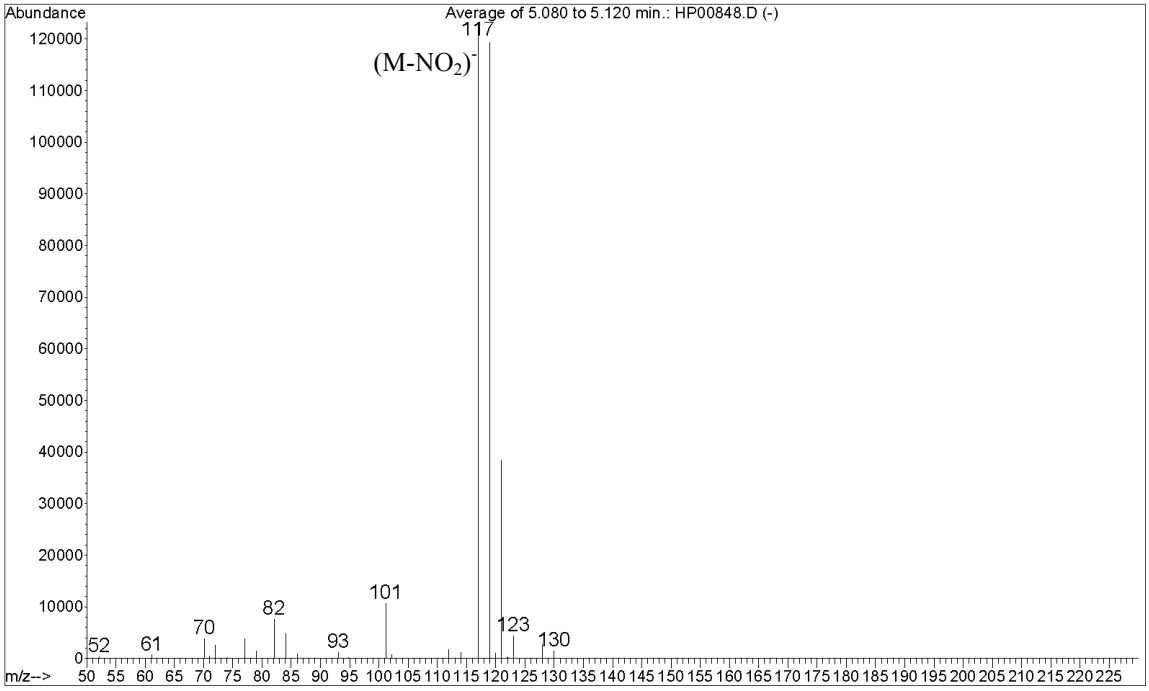
CI chromatograms supporting identification of compound **1**; TIC on left; EIC (m/z **117**) on right.

Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

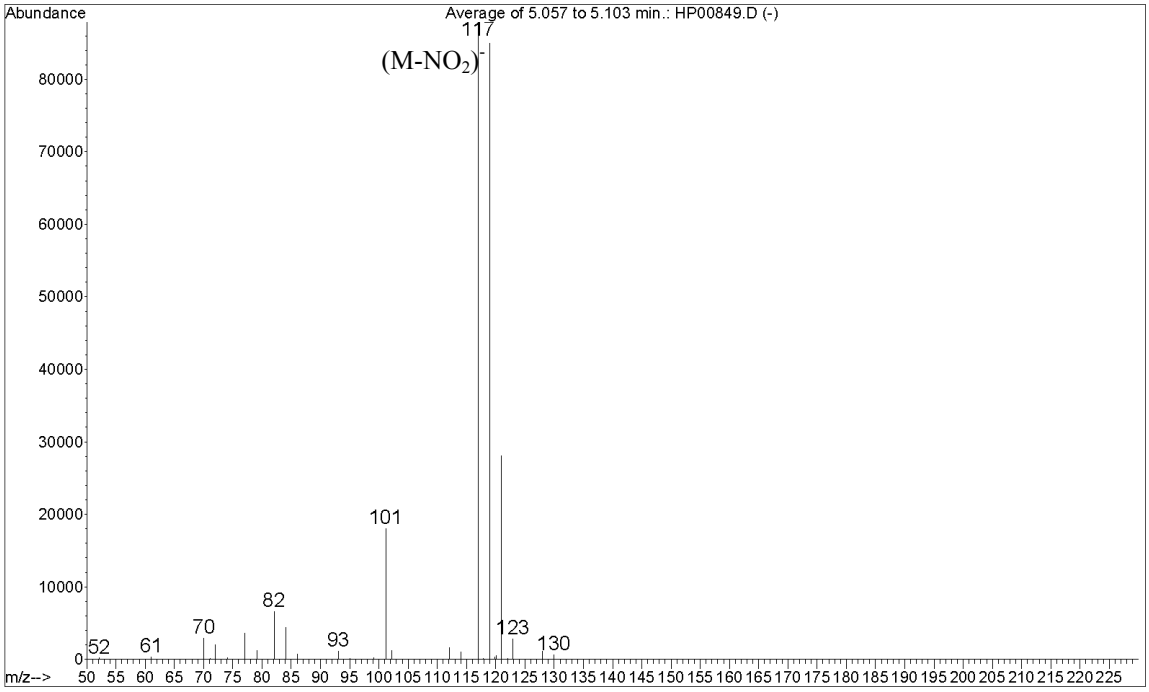
Center: Chromatograms of Organic sample, aliquot **CW-1-130-7-O** from **O/31**, retention time **5.10** min.

Bottom: Chromatograms of authentic reference standard of **Trichloronitromethane**, retention time **5.08** min.

```
File       : C:\DATA\16\HP00848.D
Acquired   : 17 Nov 2004  18:36     using AcqMethod CW-CI
Sample Name: 1uL of CW-1-130-7-O
Misc Info  : HP5-MS, 30m, 0.25 film, NCI-CH4
```




```
File       : C:\DATA\16\HP00849.D
Acquired   : 17 Nov 2004  19:23     using AcqMethod CW-CI
Sample Name: 1uL of CW-CK-1-124-2-A (compound A)
Misc Info  : HP5-MS, 30m, 0.25 film, NCI-CH4
```



CI mass spectrum of:

Top: Compound **1** in Organic sample **O/31**, aliquot **CW-1-130-7-O**

Bottom: Authentic reference standard of **Trichloronitromethane** corresponding to compound **1** (MW: **163**)

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** O/31, OB/31 **Compound number:** 2

Aliquot codes:			
Sample:	**CW-1-130-7-O**	**Blank:**	**CW-1-130-8-OB**

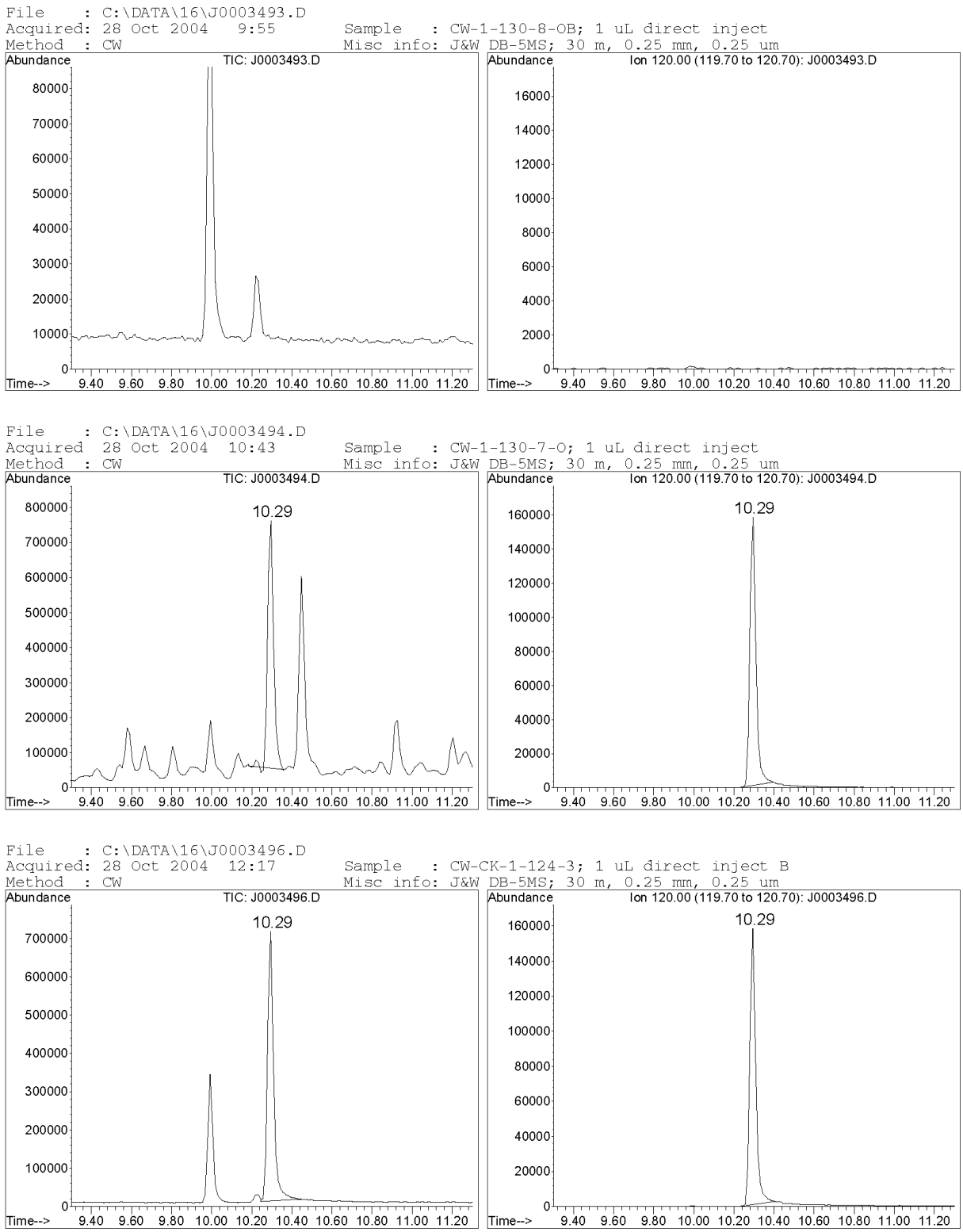
GC-EI-MS Method name:	**CW**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	3 min	**Scan range:**	30-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☒ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



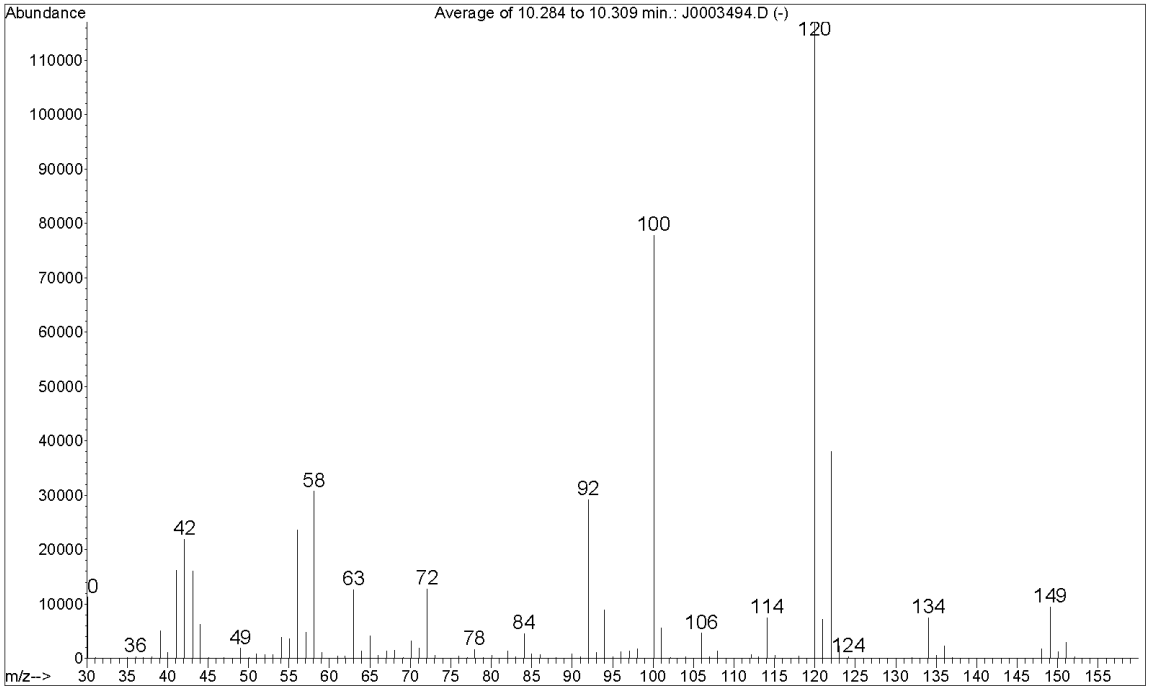
EI chromatograms supporting identification of compound **2**; TIC on left; EIC (m/z **120**) on right.

Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

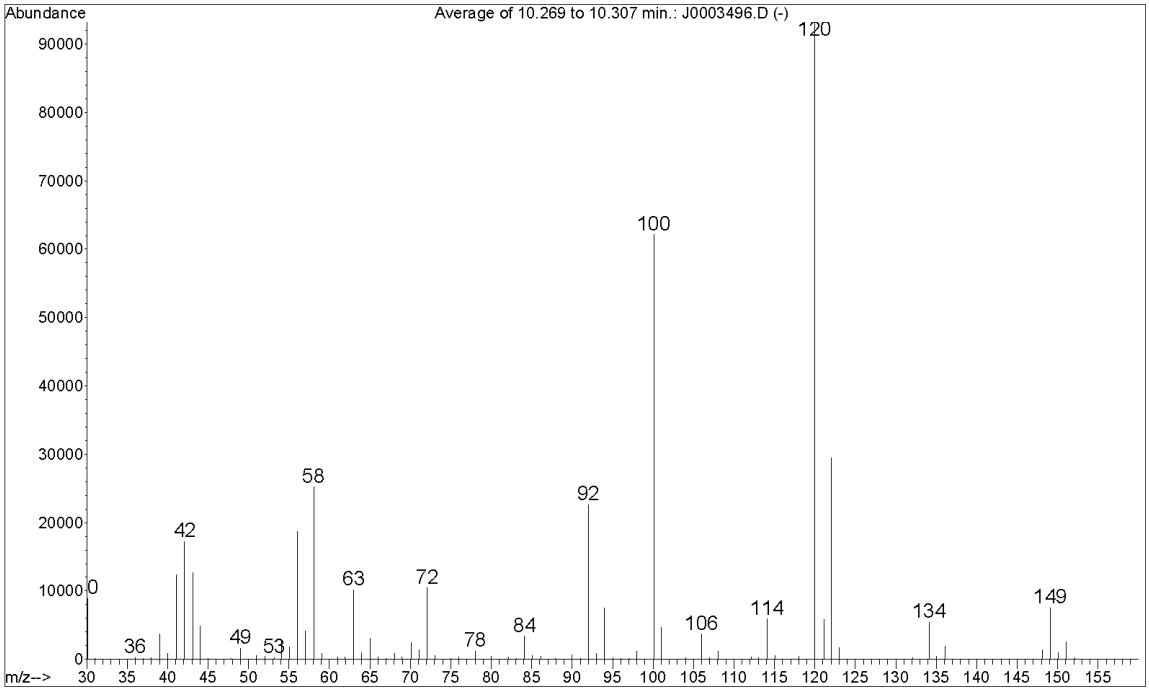
Center: Chromatograms of Organic sample, aliquot **CW-1-130-7-O** from **O/31**, retention time **10.29** min.

Bottom: Chromatograms of authentic reference standard of **2-(N-Ethyl-N-propylamino)ethylchloride**, retention time **10.29** min.

```
File       : C:\DATA\16\J0003494.D
Acquired   : 28 Oct 2004  10:43     using AcqMethod CW
Sample Name: CW-1-130-7-O; 1 uL direct inject
Misc Info  : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um
```




```
File       : C:\DATA\16\J0003496.D
Acquired   : 28 Oct 2004  12:17     using AcqMethod CW
Sample Name: CW-CK-1-124-3; 1 uL direct inject B
Misc Info  : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um
```



EI mass spectrum of:

Top: Compound **2** in Organic sample **O/31**, aliquot **CW-1-130-7-O**

Bottom: Authentic reference standard of **2-(N-Ethyl-N-propylamino)ethylchloride** corresponding to compound **2** (MW: **149**)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** O/31, OB/31 **Compound number:** 2

Aliquot codes:			
Sample:	**CW-1-130-7-O**	**Blank:**	**CW-1-130-8-OB**

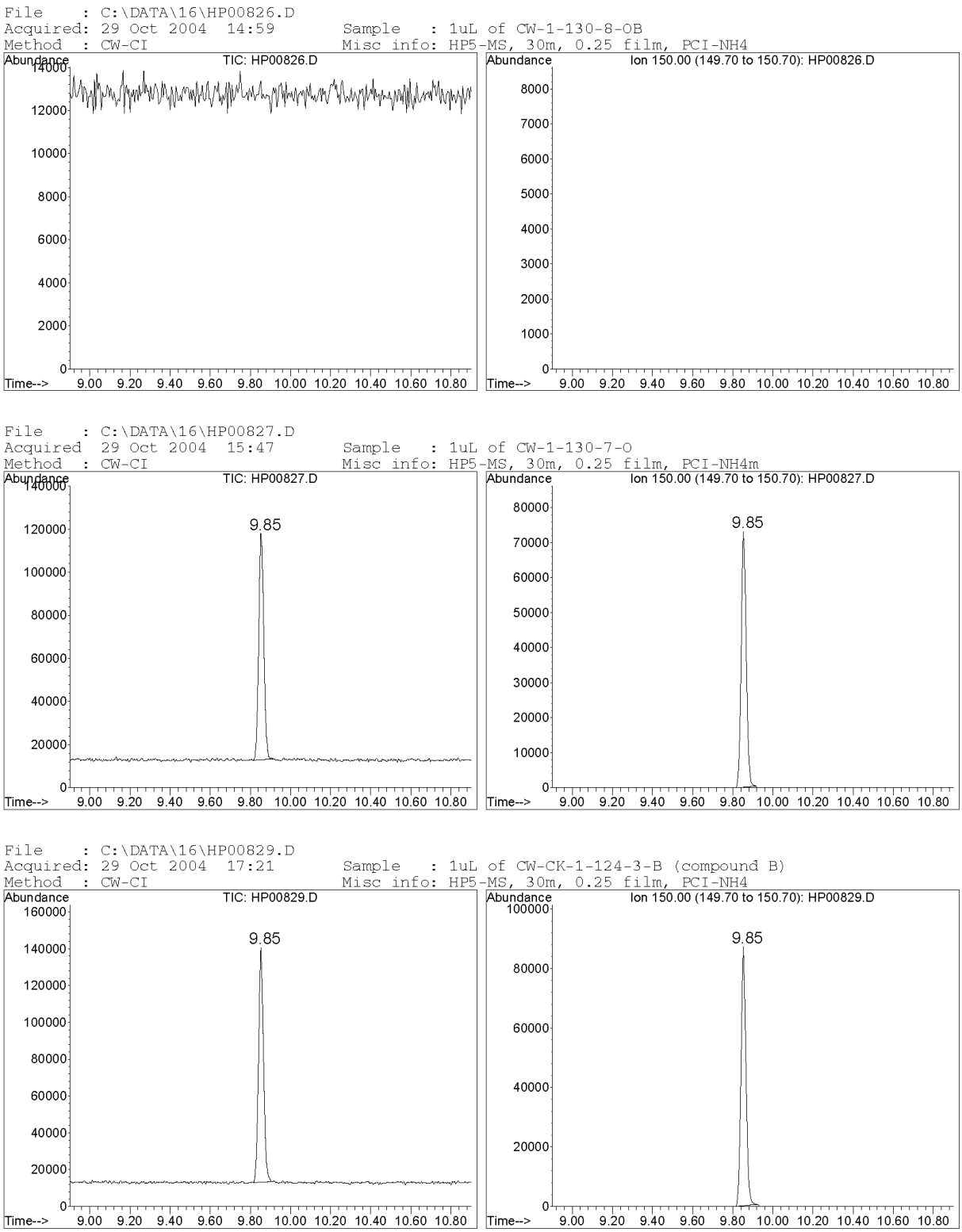
GC-CI-MS Method name:	**CW-CI**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☐ Methane ☐ Isobutane ☒ Ammonia ☐ Other:		
Solvent delay time:	3 min	**Scan range:**	50-550 m/z
Electron energy:	235 eV	**Scan time:**	0.35 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☒ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



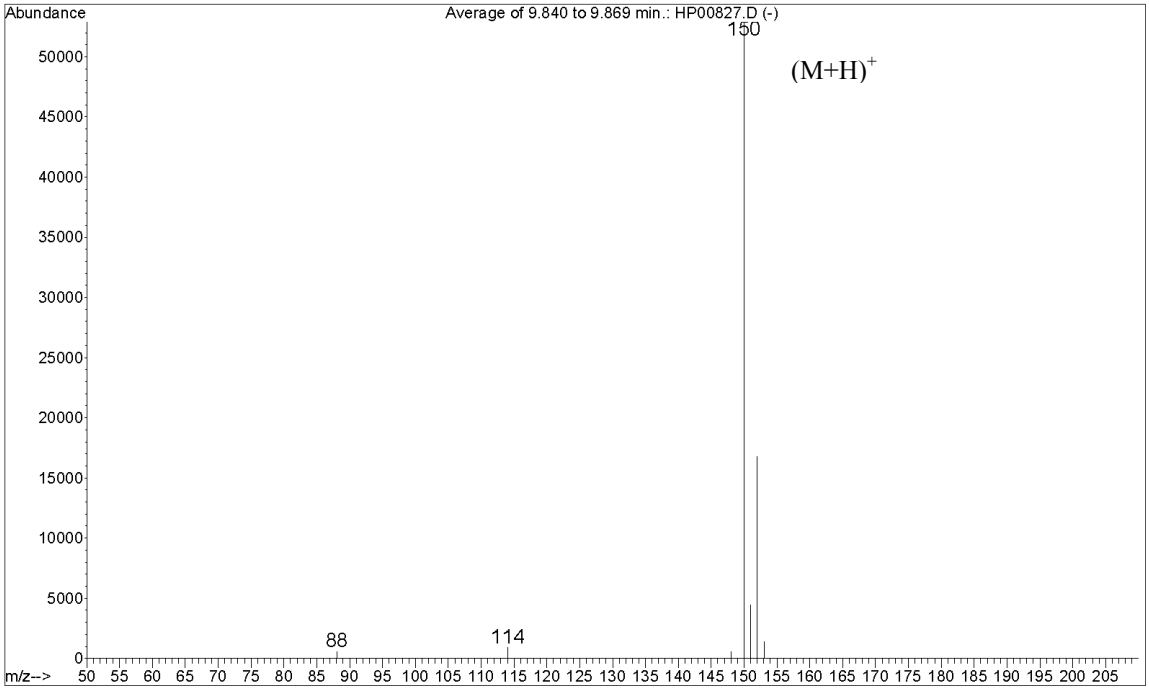
CI chromatograms supporting identification of compound **2**; TIC on left; EIC (m/z **150**) on right.

Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

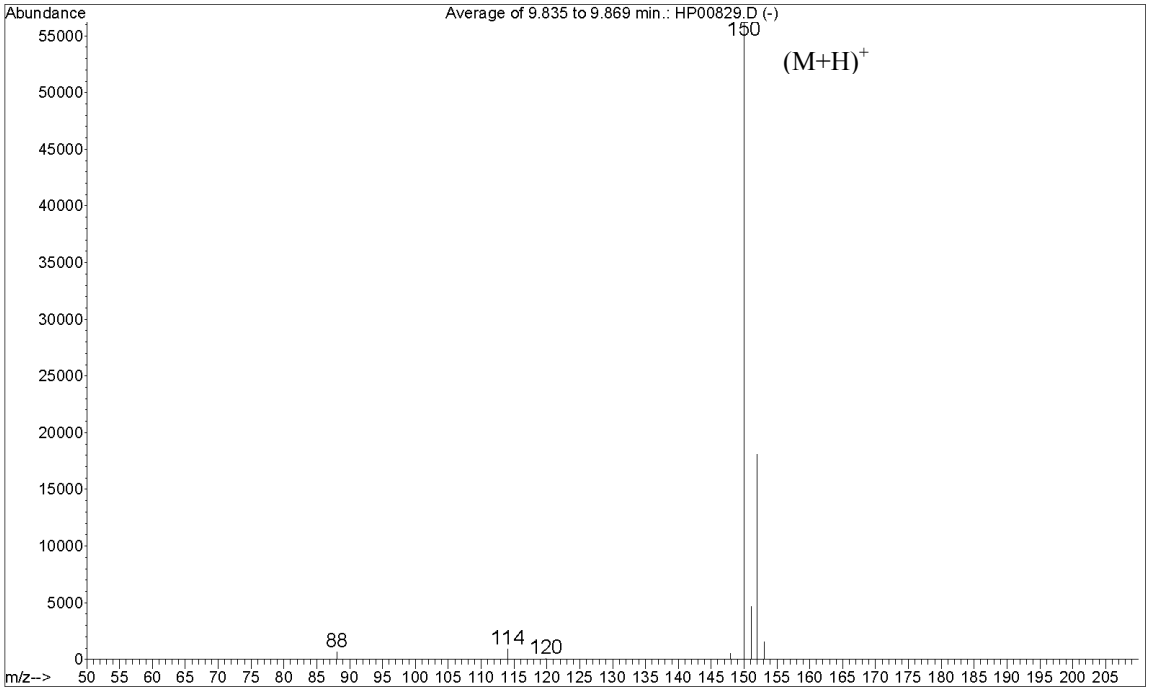
Center: Chromatograms of Organic sample, aliquot **CW-1-130-7-O** from **O/31**, retention time **9.85** min.

Bottom: Chromatograms of authentic reference standard of **2-(N-Ethyl-N-propylamino)ethylchloride**, retention time **9.85** min.

```
File       : C:\DATA\16\HP00827.D
Acquired   : 29 Oct 2004  15:47     using AcqMethod CW-CI
Sample Name: 1uL of CW-1-130-7-O
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH4m
```




```
File       : C:\DATA\16\HP00829.D
Acquired   : 29 Oct 2004  17:21     using AcqMethod CW-CI
Sample Name: 1uL of CW-CK-1-124-3-B (compound B)
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH4
```



CI mass spectrum of:

Top: Compound **2** in Organic sample **O/31**, aliquot **CW-1-130-7-O**

Bottom: Authentic reference standard of **2-(N-Ethyl-N-propylamino)ethylchloride** corresponding to compound **2** (MW: **149**)

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** O/31, OB/31 **Compound number:** 3

Aliquot codes:			
Sample:	**CW-1-130-7-O**	**Blank:**	**CW-1-130-8-OB**

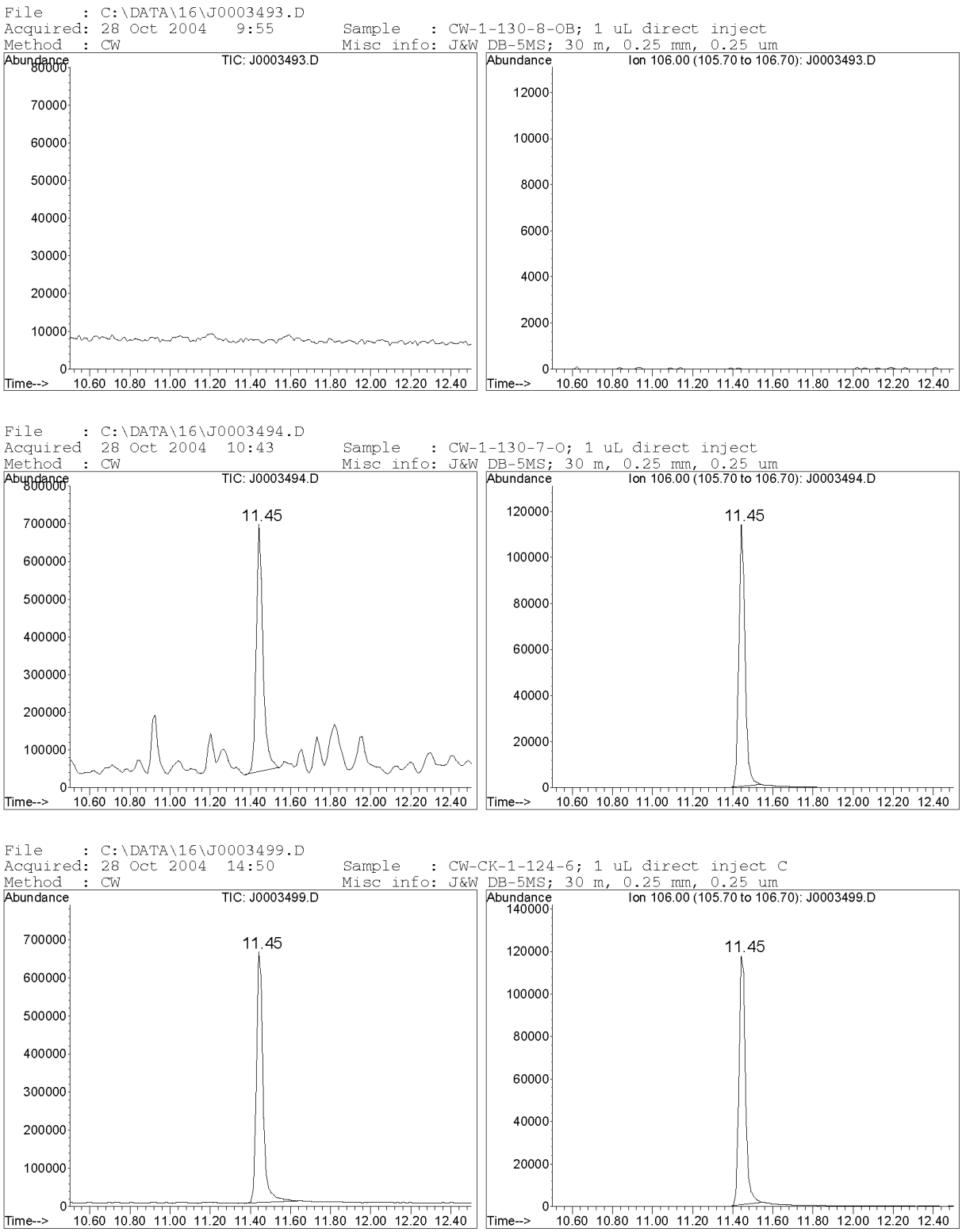
GC-EI-MS Method name:	**CW**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	3 min	**Scan range:**	30-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



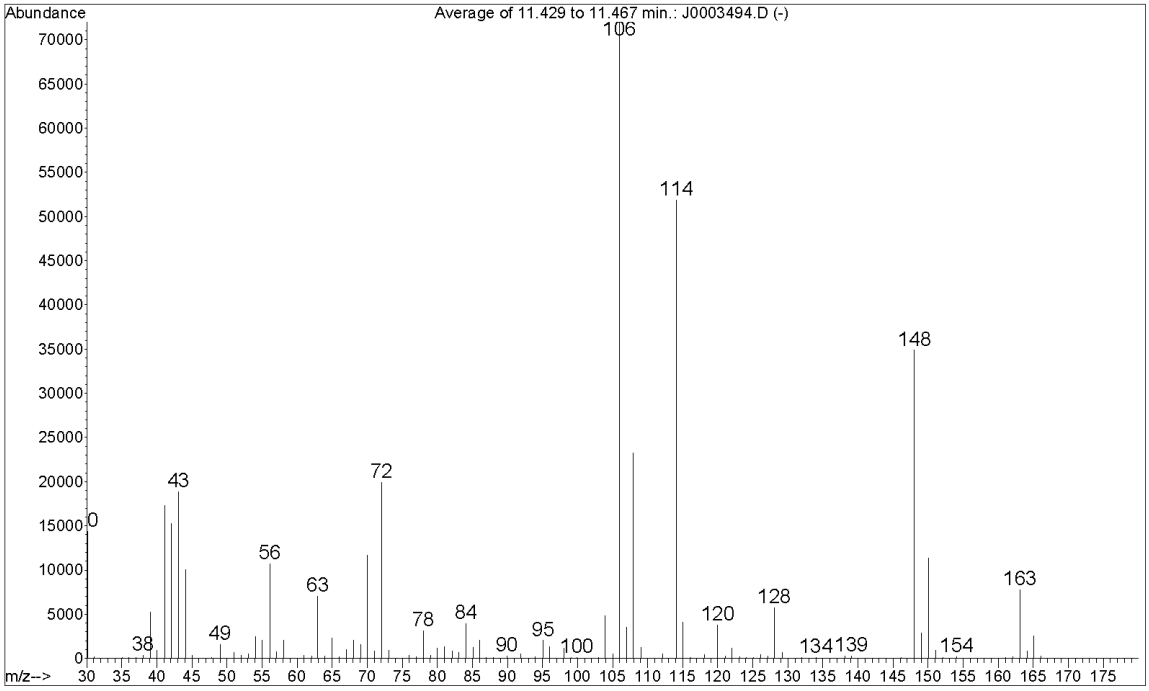
EI chromatograms supporting identification of compound **3**; TIC on left; EIC (m/z **106**) on right.

Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

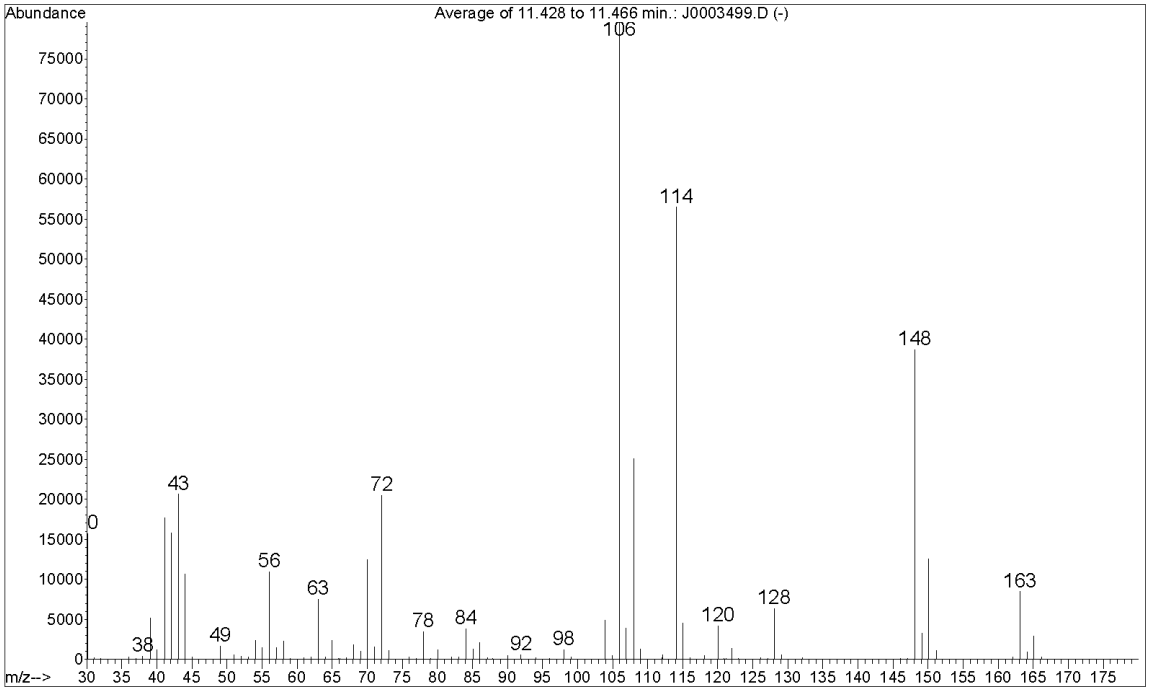
Center: Chromatograms of Organic sample, aliquot **CW-1-130-7-O** from **O/31**, retention time **11.45** min.

Bottom: Chromatograms of authentic reference standard of **2-(N,N-Diisopropylamino)ethylchloride**, retention time **11.45** min.

```
File       : C:\DATA\16\J0003494.D
Acquired   : 28 Oct 2004  10:43     using AcqMethod CW
Sample Name: CW-1-130-7-O; 1 uL direct inject
Misc Info  : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um
```




```
File       : C:\DATA\16\J0003499.D
Acquired   : 28 Oct 2004  14:50     using AcqMethod CW
Sample Name: CW-CK-1-124-6; 1 uL direct inject C
Misc Info  : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um
```



EI mass spectrum of:

Top: Compound **3** in Organic sample **O/31**, aliquot **CW-1-130-7-O**

Bottom: Authentic reference standard of **2-(N,N-Diisopropylamino)ethylchloride** corresponding to compound **3** (MW: **163**)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** O/31, OB/31 **Compound number:** 3

Aliquot codes:			
Sample:	**CW-1-130-7-O**	**Blank:**	**CW-1-130-8-OB**

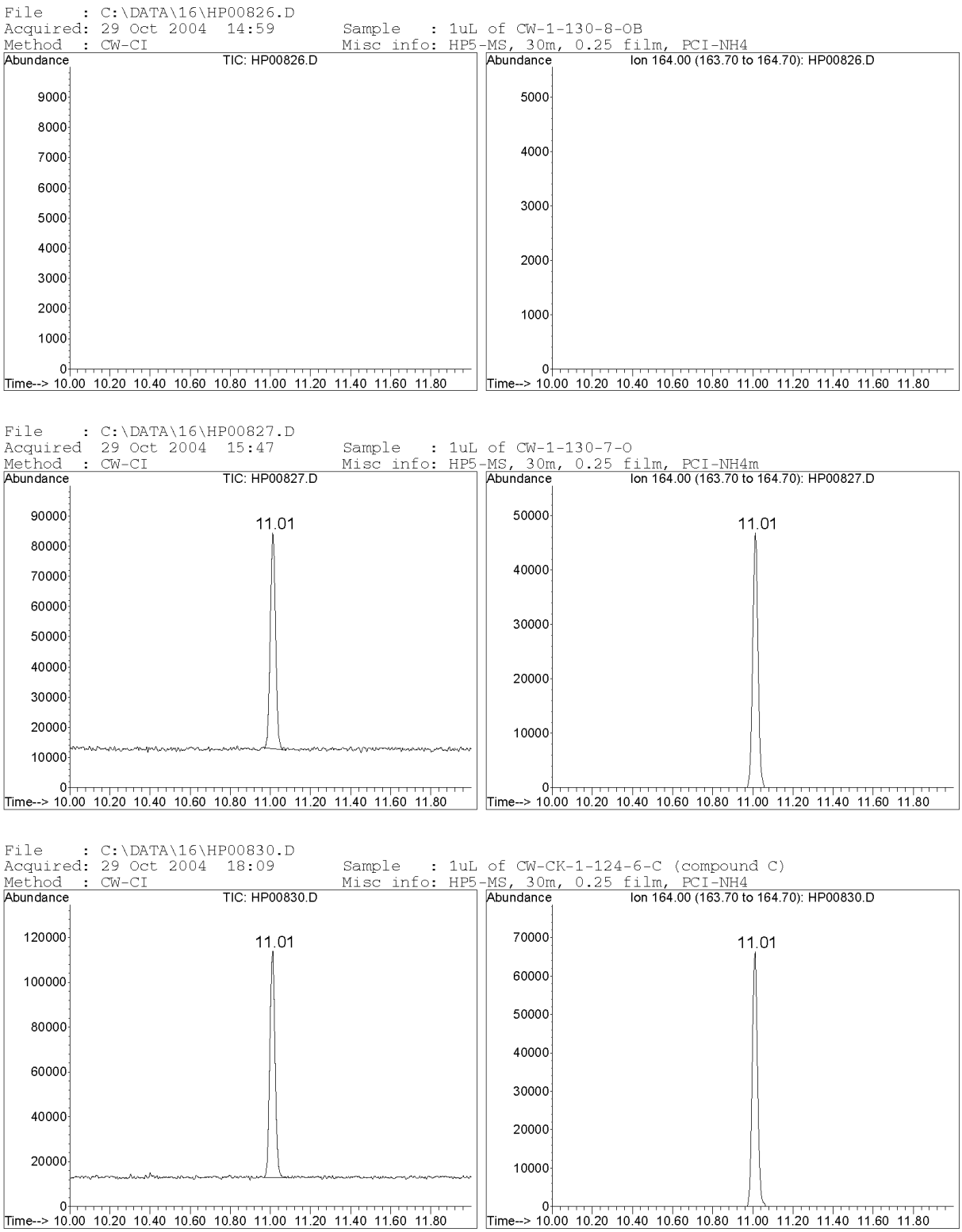
GC-CI-MS Method name:	**CW-CI**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☐ Methane ☐ Isobutane ☒ Ammonia ☐ Other:		
Solvent delay time:	3 min	**Scan range:**	50-550 m/z
Electron energy:	235 eV	**Scan time:**	0.35 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



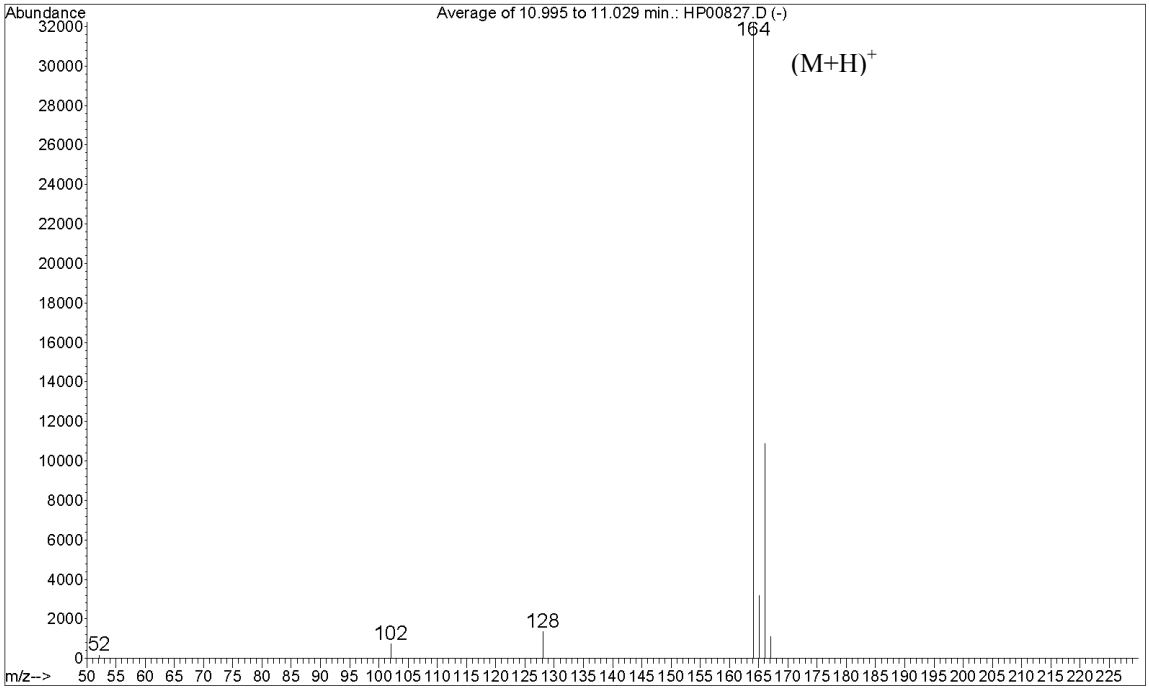
CI chromatograms supporting identification of compound **3**; TIC on left; EIC (m/z **164**) on right.

Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

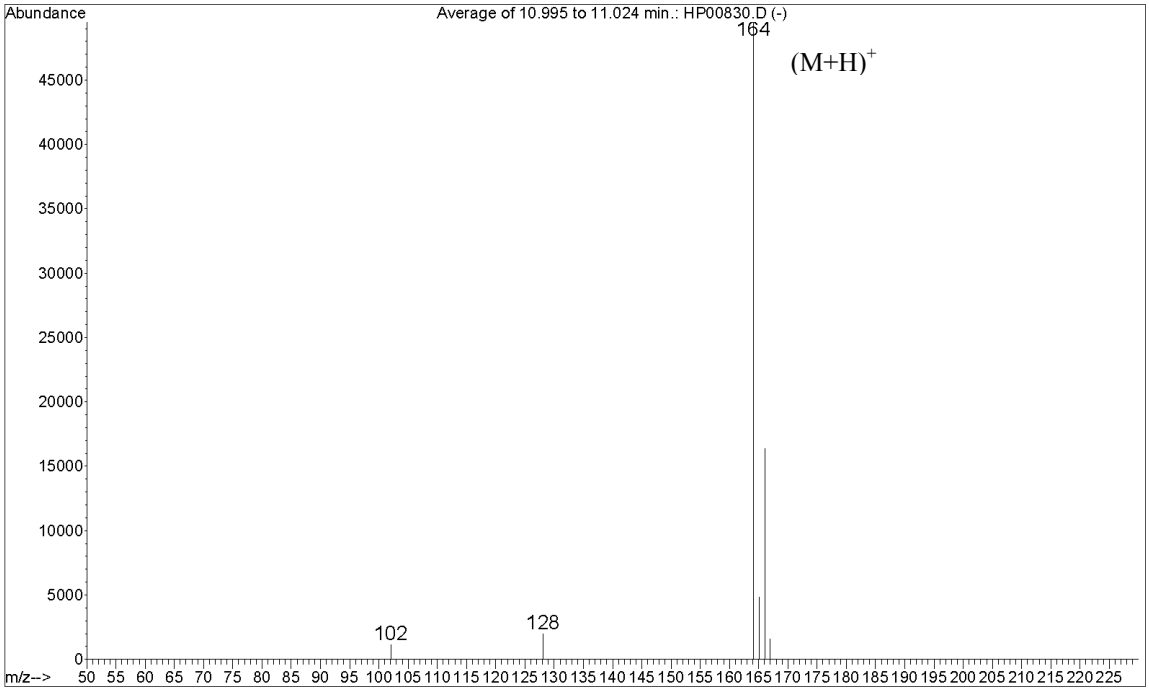
Center: Chromatograms of Organic sample, aliquot **CW-1-130-7-O** from **O/31**, retention time **11.01** min.

Bottom: Chromatograms of authentic reference standard of **2-(N,N-Diisopropylamino)ethylchloride**, retention time **11.01** min.

```
File       : C:\DATA\16\HP00827.D
Acquired   : 29 Oct 2004  15:47     using AcqMethod CW-CI
Sample Name: 1uL of CW-1-130-7-O
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH4m
```




```
File       : C:\DATA\16\HP00830.D
Acquired   : 29 Oct 2004  18:09     using AcqMethod CW-CI
Sample Name: 1uL of CW-CK-1-124-6-C (compound C)
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH4
```



CI mass spectrum of:

Top: Compound **3** in Organic sample **O/31**, aliquot **CW-1-130-7-O**

Bottom: Authentic reference standard of **2-(N,N-Diisopropylamino)ethylchloride** corresponding to compound **3** (MW: **163**)

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** O/31, OB/31 **Compound number:** 4

Aliquot codes:			
Sample:	**CW-1-130-7-O**	**Blank:**	**CW-1-130-8-OB**

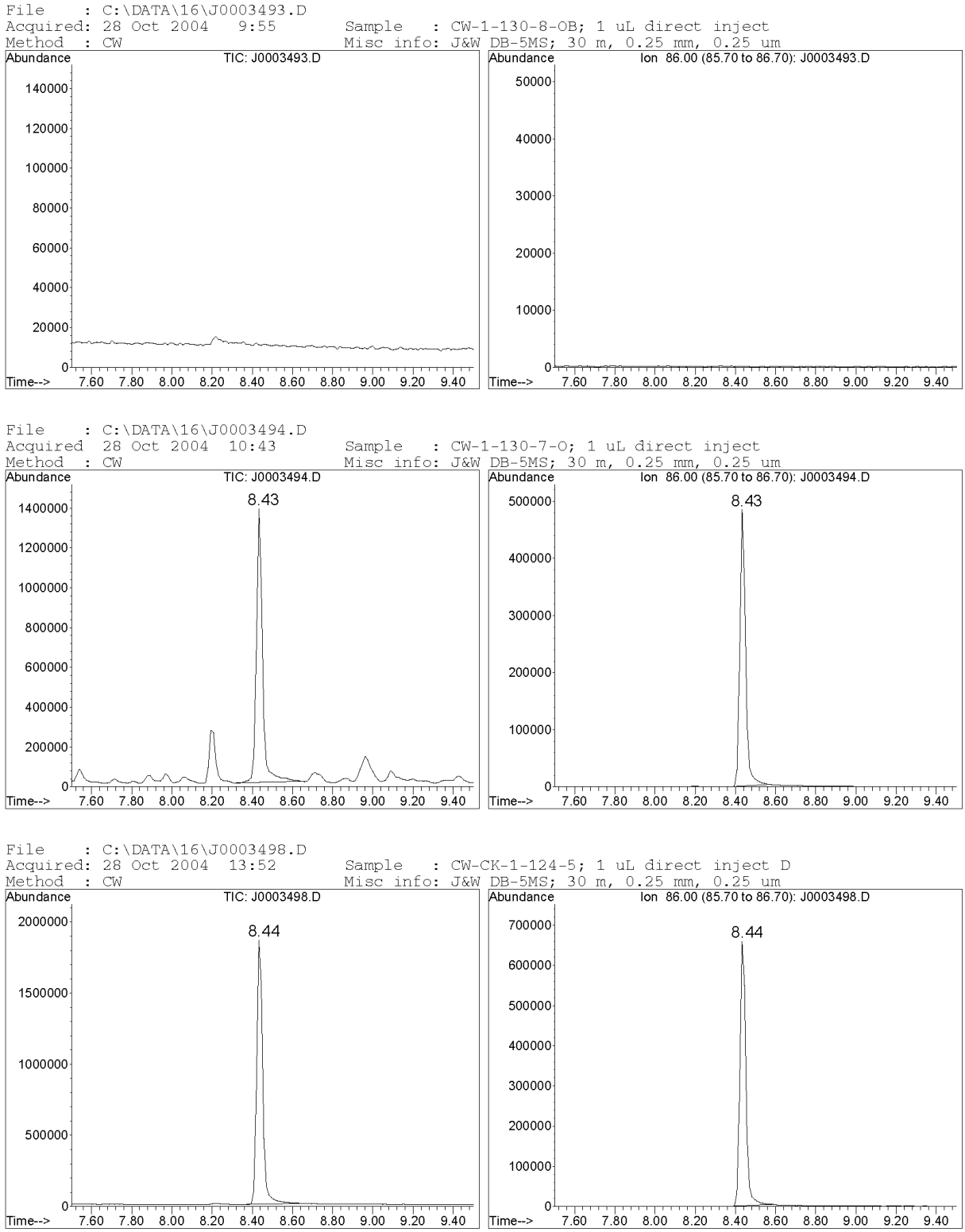
GC-EI-MS Method name:	**CW**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	3 min	**Scan range:**	30-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



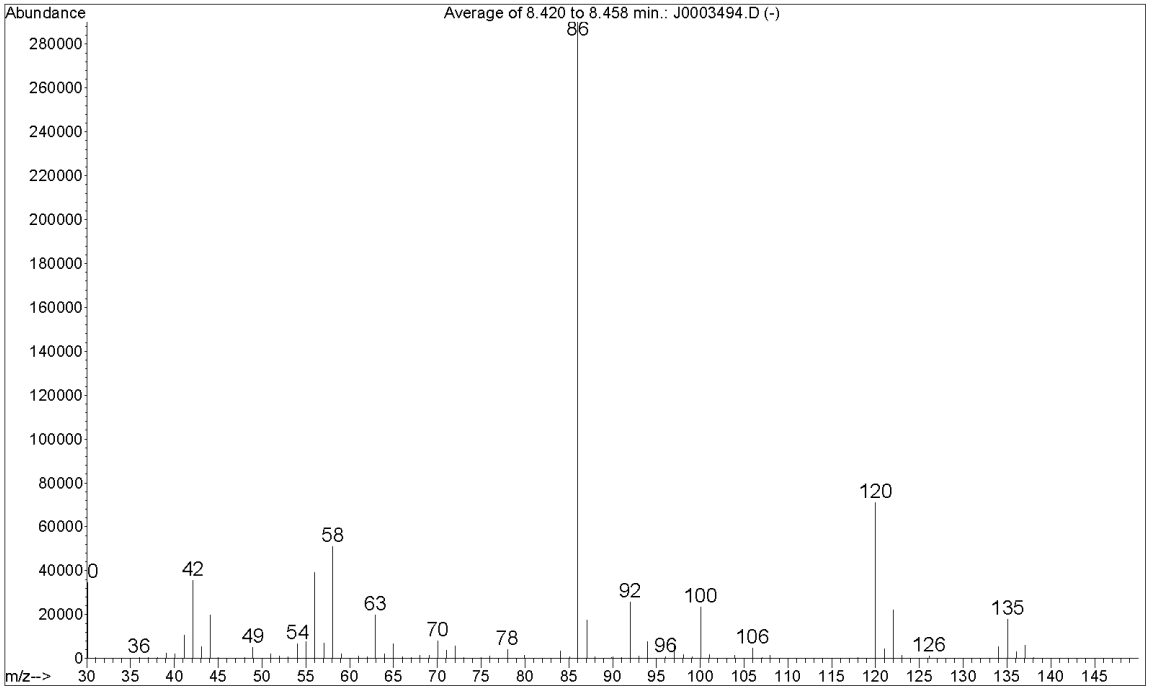
EI chromatograms supporting identification of compound **4**; TIC on left; EIC (m/z **86**) on right.

Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

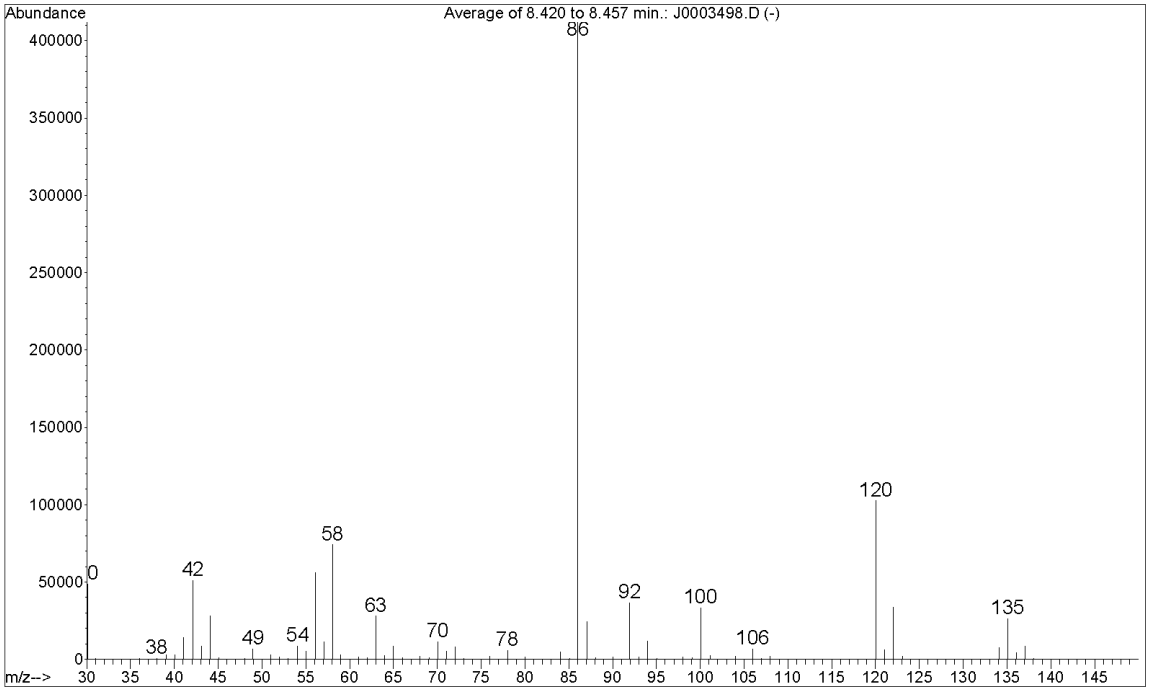
Center: Chromatograms of Organic sample, aliquot **CW-1-130-7-O** from **O/31**, retention time **8.43** min.

Bottom: Chromatograms of authentic reference standard of **2-(N,N-Diethylamino)ethylchloride**, retention time **8.44** min.

```
File       : C:\DATA\16\J0003494.D
Acquired   : 28 Oct 2004  10:43     using AcqMethod CW
Sample Name: CW-1-130-7-O; 1 uL direct inject
Misc Info  : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um
```




```
File       : C:\DATA\16\J0003498.D
Acquired   : 28 Oct 2004  13:52     using AcqMethod CW
Sample Name: CW-CK-1-124-5; 1 uL direct inject D
Misc Info  : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um
```



EI mass spectrum of:

Top: Compound **4** in Organic sample **O/31**, aliquot **CW-1-130-7-O**

Bottom: Authentic reference standard of **2-(N,N-Diethylamino)ethylchloride** corresponding to compound **4** (MW: **135**)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** O/31, OB/31 **Compound number:** 4

Aliquot codes:			
Sample:	**CW-1-130-7-O**	**Blank:**	**CW-1-130-8-OB**

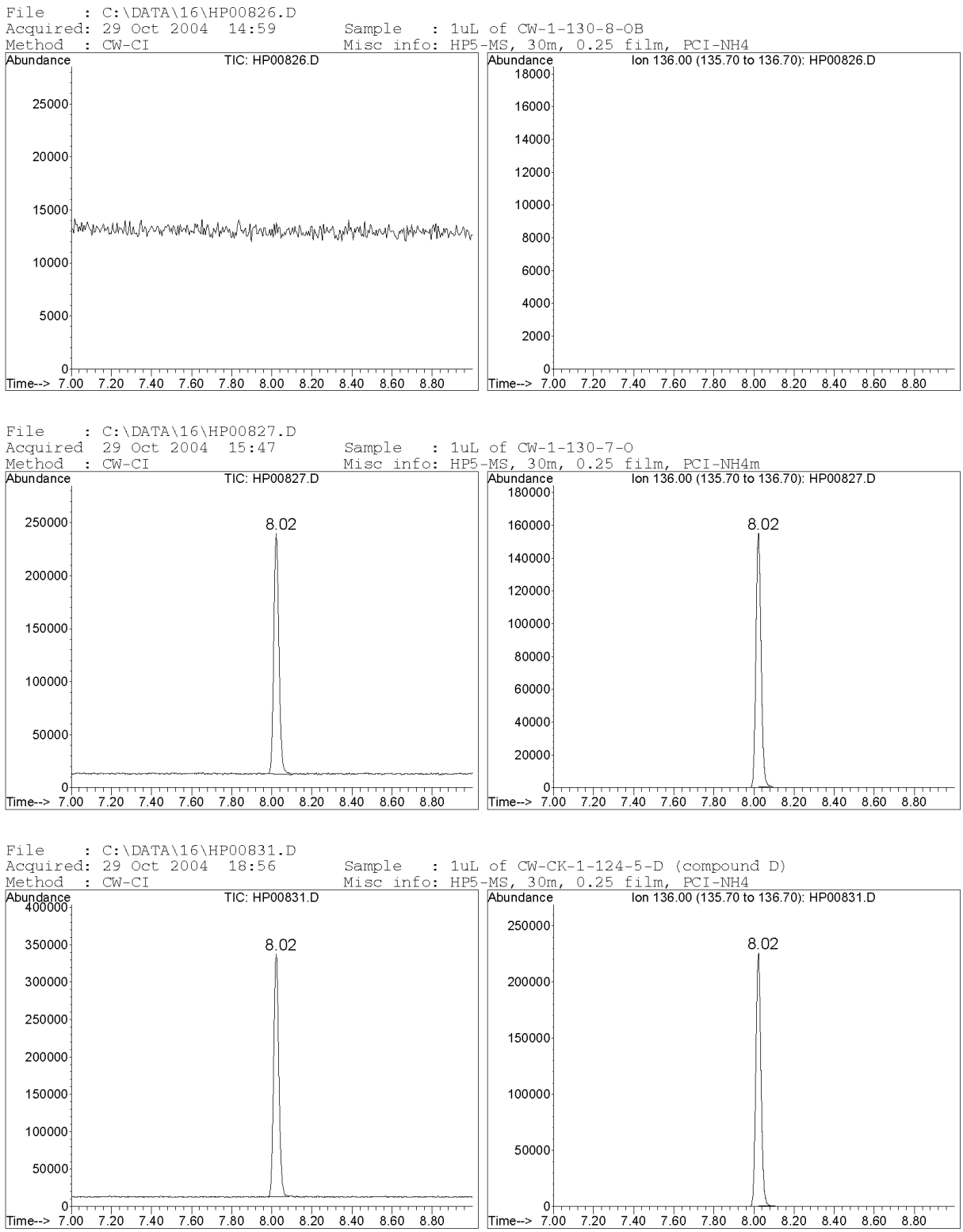
GC-CI-MS Method name:	**CW-CI**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☐ Methane ☐ Isobutane ☒ Ammonia ☐ Other:		
Solvent delay time:	3 min	**Scan range:**	50-550 m/z
Electron energy:	235 eV	**Scan time:**	0.35 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



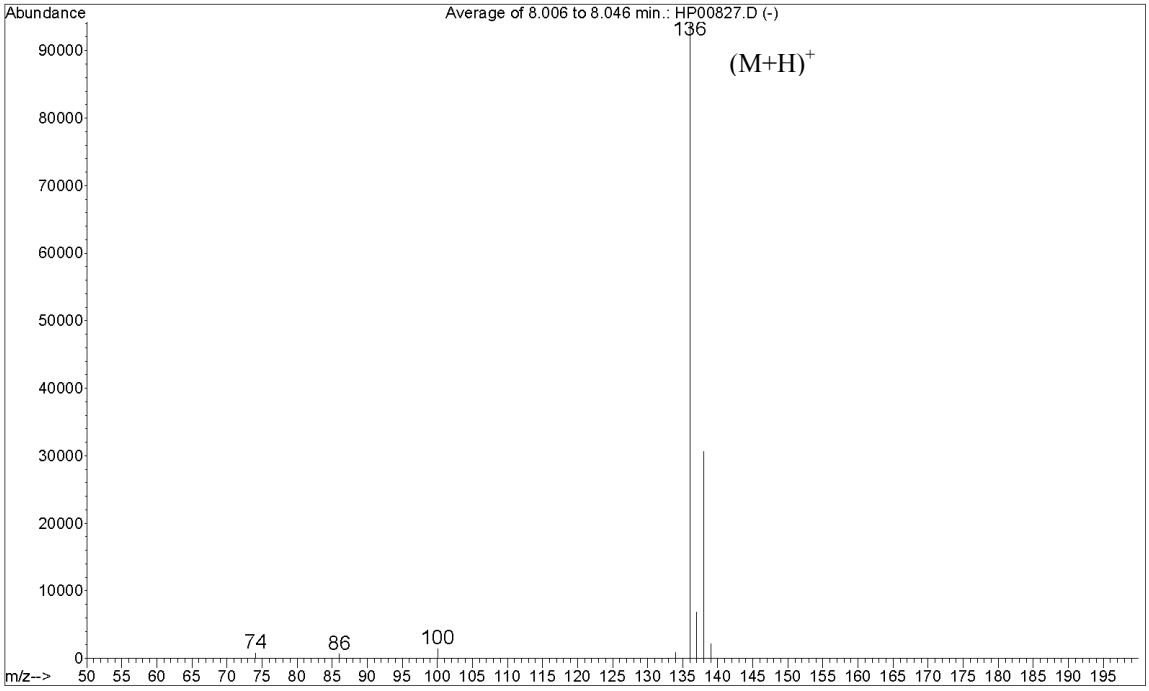
CI chromatograms supporting identification of compound **4**; TIC on left; EIC (m/z **136**) on right.

Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

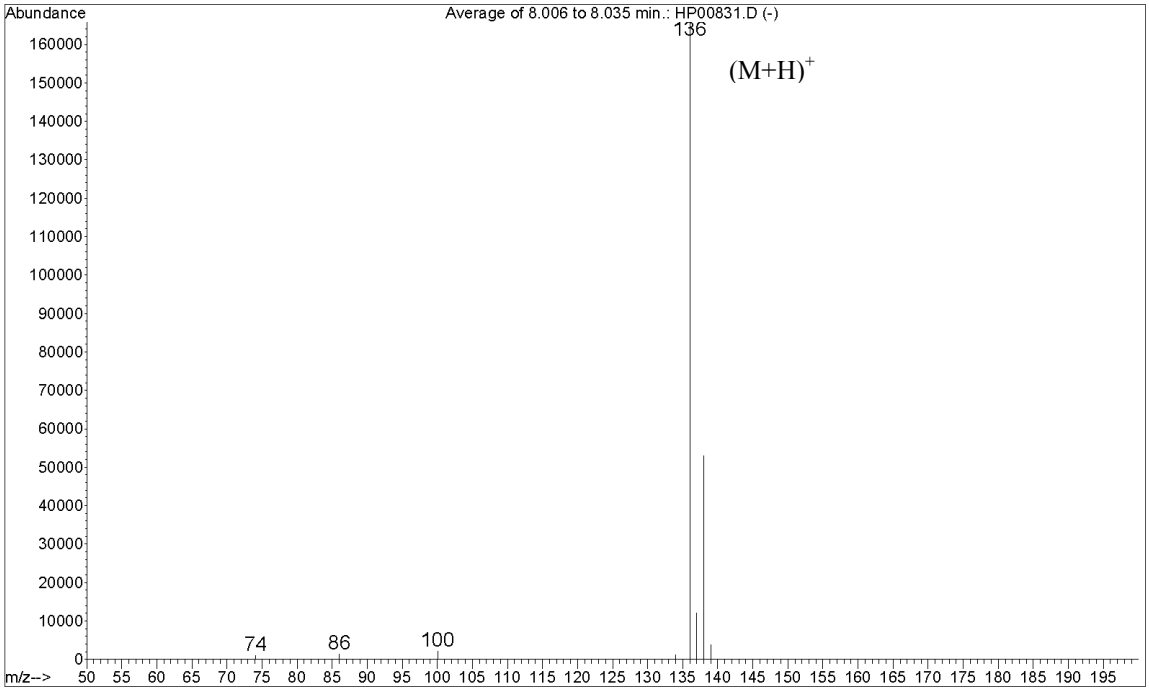
Center: Chromatograms of Organic sample, aliquot **CW-1-130-7-O** from **O/31**, retention time **8.02** min.

Bottom: Chromatograms of authentic reference standard of **2-(N,N-Diethylamino)ethylchloride**, retention time **8.02** min.

```
File       : C:\DATA\16\HP00827.D
Acquired   : 29 Oct 2004  15:47     using AcqMethod CW-CI
Sample Name: 1uL of CW-1-130-7-O
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH4m
```




```
File       : C:\DATA\16\HP00831.D
Acquired   : 29 Oct 2004  18:56     using AcqMethod CW-CI
Sample Name: 1uL of CW-CK-1-124-5-D (compound D)
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH4
```



CI mass spectrum of:

Top: Compound **4** in Organic sample **O/31**, aliquot **CW-1-130-7-O**

Bottom: Authentic reference standard of **2-(N,N-Diethylamino)ethylchloride** corresponding to compound **4** (MW: **135**)

SAMPLE PREPARATION DESCRIPTION

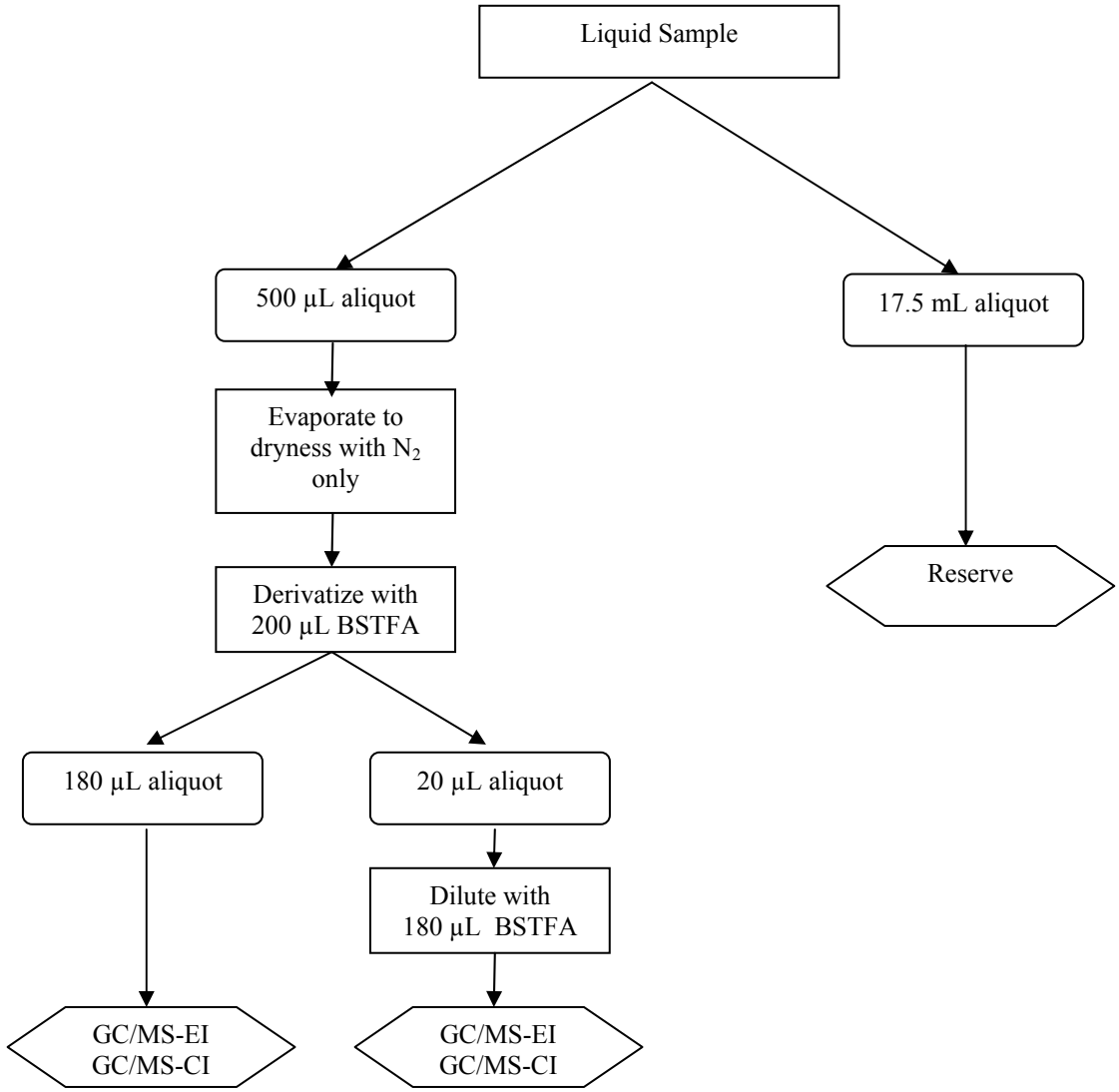
Laboratory code: 31 Sample code(s): L/31 Sample Blank code: LB/31

1. Sample preparation

Sample/ Aliquot Code	*Specification of Sample/ Type of Sample Preparation*	**Amount/ Volume**	*Sample Preparation Procedures*	**End Volume**	**Resulting Aliquot Code**	**Analytical Technique(s)**
L/31 LB/31	TMS derivative of sample	500 µL	500 µL evaporated to dryness (N_2, no heat). Added 500 µL BSTFA. Reacted at 60°C for 30 minutes.	500 µL	CW-1-131-4-L CW-1-131-3-LB	GC/MS-EI GC/MS-CI
CW-1-131-4-L	Dilution	20 µL	Diluted TMS derivitized samples with 180 µL BSTFA.	200 µL	CW-1-131-5-L	GC/MS-EI GC/MS-CI

2. Additional information

Note: sample CW-1-131-4-L was diluted with the same batch of BSTFA with which it had been derivitized to produce sample CW-1-131-5-L. Blank CW-1-131-3-LB was used directly as a blank (worst case – any scheduled compounds would be seen 10x as compared to the diluted sample).



Note: This flowchart is for visualization only; see the preceding sample preparation description page for sample aliquot numbers

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** L/31, LB/31 **Compound number:** 5

Aliquot codes:			
Sample:	**CW-1-131-5-L**	**Blank:**	**CW-1-131-3-LB**

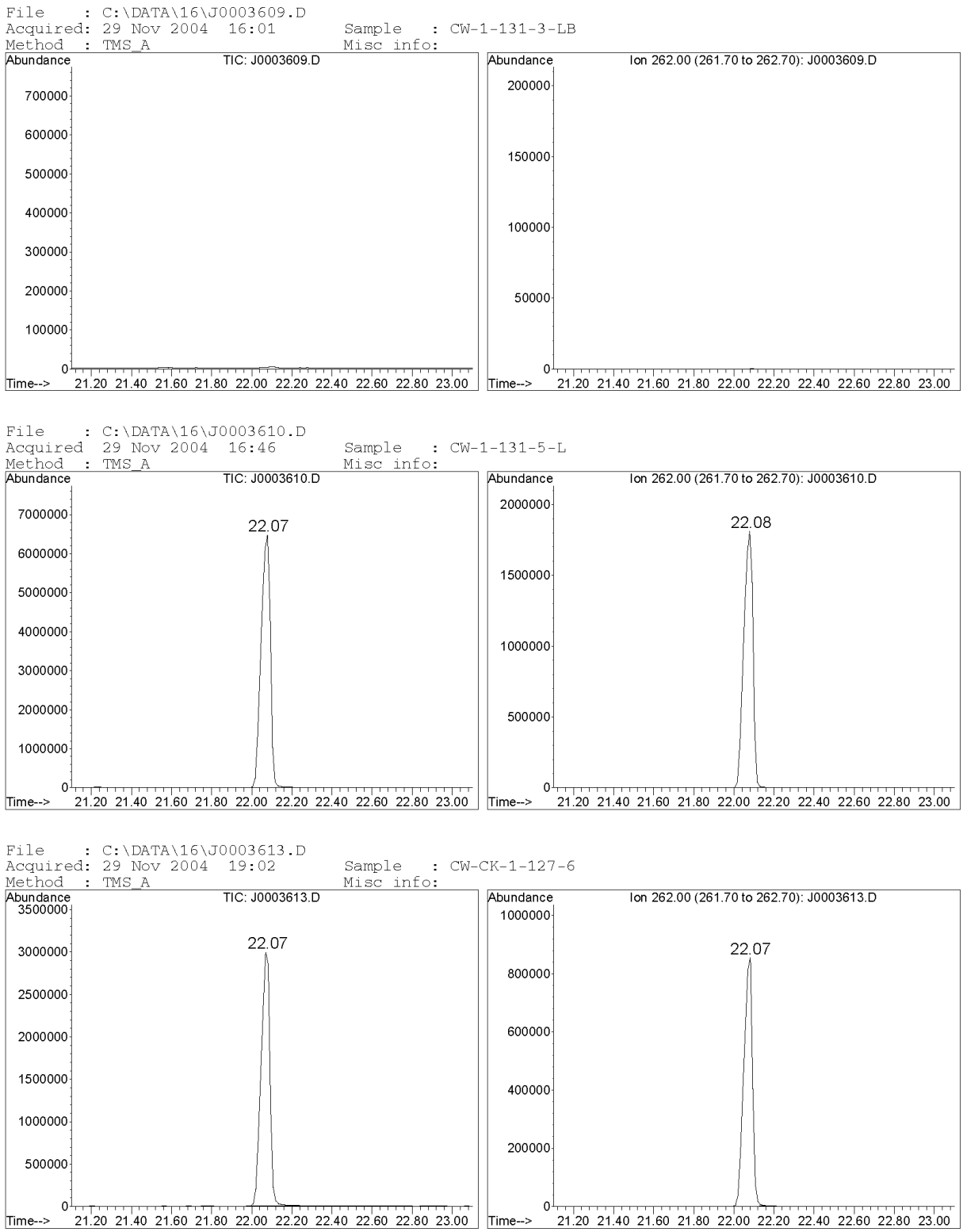
GC-EI-MS Method name:	**TMS_A**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 38 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0. 70 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	70 °C (8 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	8 min	**Scan range:**	40-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☐ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☒ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



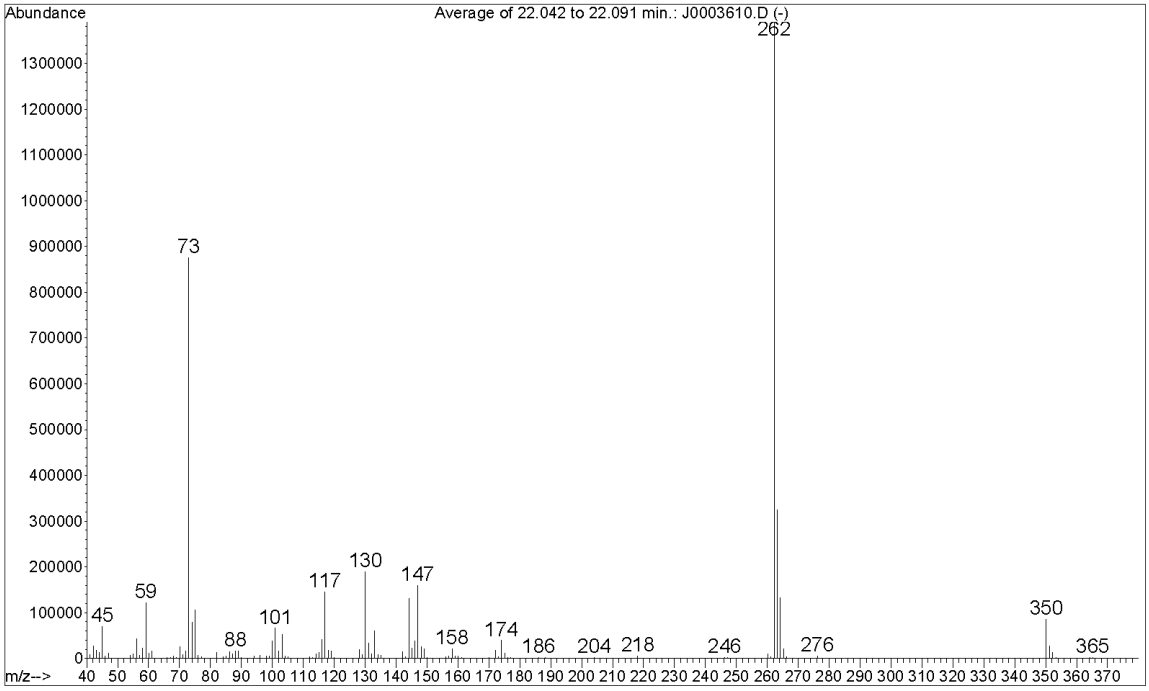
EI chromatograms supporting identification of compound **5**; TIC on left; EIC (m/z **262**) on right.

Top: Chromatograms of Liquid blank, aliquot **CW-1-131-3-LB** from **LB/31**.

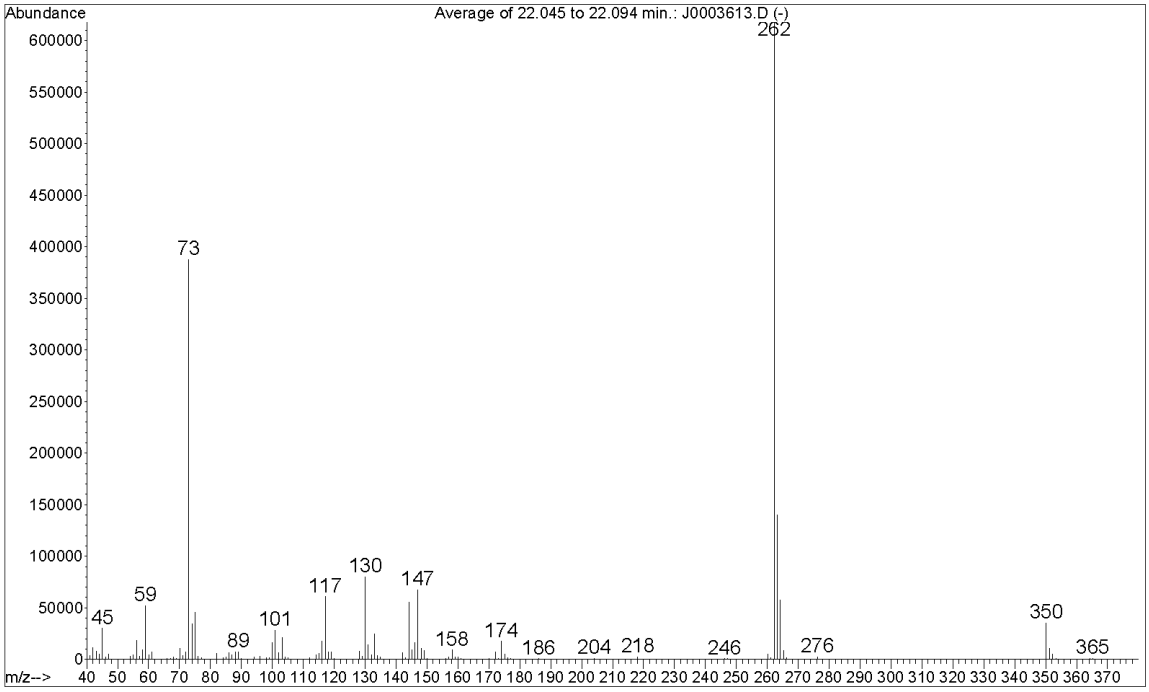
Center: Chromatograms of Liquid sample, aliquot **CW-1-131-5-L** from **L/31**, retention time **22.07** min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Triethanolamine** [tris(2-trimethylsiloxyethyl)amine], retention time **22.07** min.

```
File       : C:\DATA\16\J0003610.D
Acquired   : 29 Nov 2004  16:46     using AcqMethod TMS_A
Sample Name: CW-1-131-5-L
Misc Info  :
```




```
File       : C:\DATA\16\J0003613.D
Acquired   : 29 Nov 2004  19:02     using AcqMethod TMS_A
Sample Name: CW-CK-1-127-6
Misc Info  :
```



EI mass spectrum of:

Top: Compound **5** in Liquid sample **L/31**, aliquot **CW-1-131-5-L**

Bottom: TMS derivative of the authentic reference standard of **Triethanolamine** [tris(2-trimethylsiloxyethyl)amine] corresponding to compound **5** (MW: **365**)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** L/31, LB/31 **Compound number:** 5

Aliquot codes:			
Sample:	**CW-1-131-5-L**	**Blank:**	**CW-1-131-3-LB**

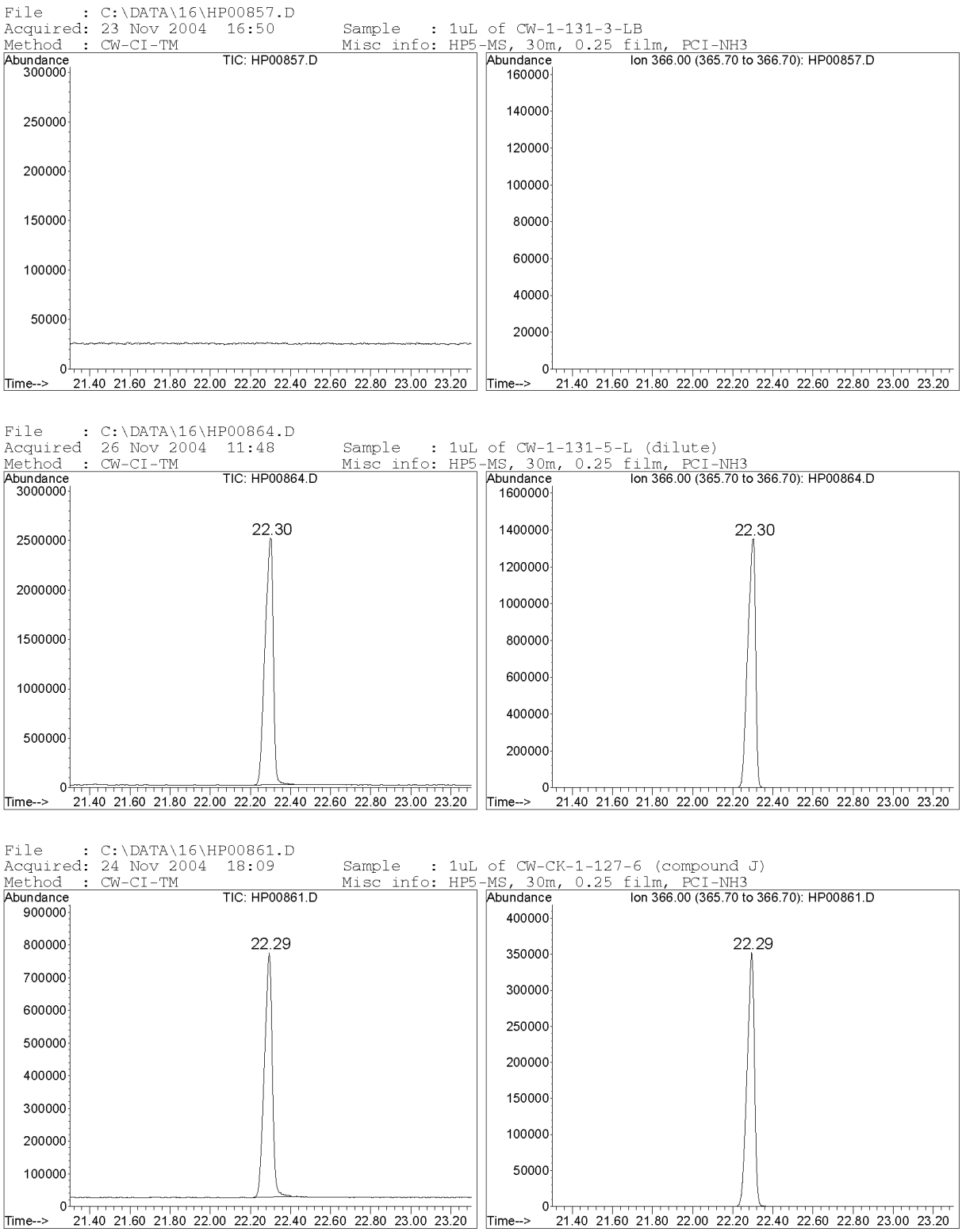
GC-CI-MS Method name:	**CW-CI-TM**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	70 °C (8 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☐ Methane ☐ Isobutane ☒ Ammonia ☐ Other:		
Solvent delay time:	8 min	**Scan range:**	50-550 m/z
Electron energy:	235 eV	**Scan time:**	0.35 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☐ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☒ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



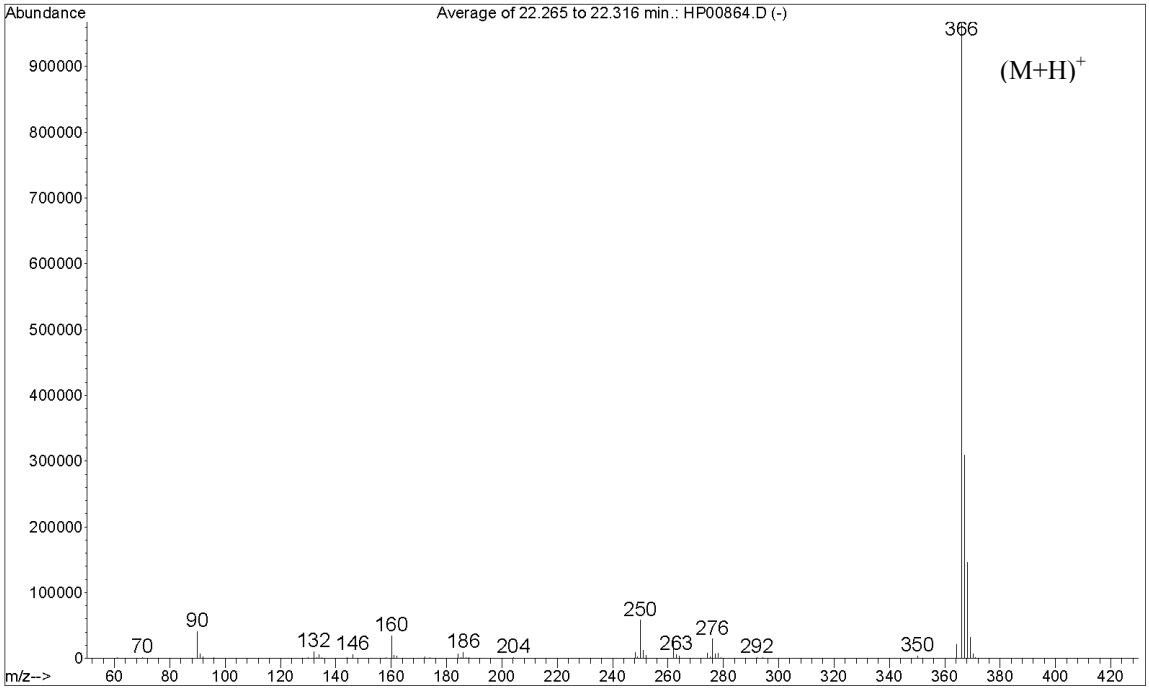
CI chromatograms supporting identification of compound **5**; TIC on left; EIC (m/z **366**) on right.

Top: Chromatograms of Liquid blank, aliquot **CW-1-131-3-LB** from **LB/31**.

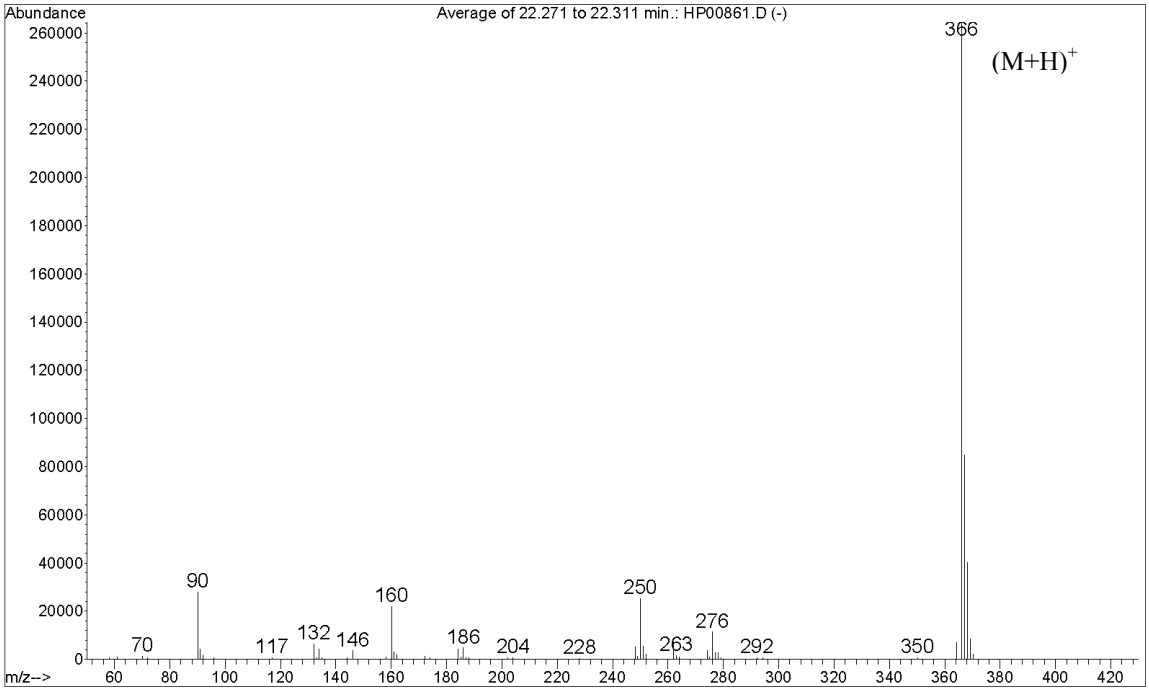
Center: Chromatograms of Liquid sample, aliquot **CW-1-131-5-L** from **L/31**, retention time **22.30** min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Triethanolamine** [tris(2-trimethylsiloxyethyl)amine], retention time **22.29** min.

```
File       : C:\DATA\16\HP00864.D
Acquired   : 26 Nov 2004  11:48     using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-1-131-5-L (dilute)
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH3
```




```
File       : C:\DATA\16\HP00861.D
Acquired   : 24 Nov 2004  18:09     using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-CK-1-127-6 (compound J)
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH3
```



CI mass spectrum of:

Top: Compound **5** in Liquid sample **L/31**, aliquot **CW-1-131-5-L**

Bottom: TMS derivative of the authentic reference standard of **Triethanolamine** [tris(2-trimethylsiloxyethyl)amine] corresponding to compound **5** (MW: **365**)

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** L/31, LB/31 **Compound number:** 6

Aliquot codes:			
Sample:	**CW-1-131-4-L**	**Blank:**	**CW-1-131-3-LB**

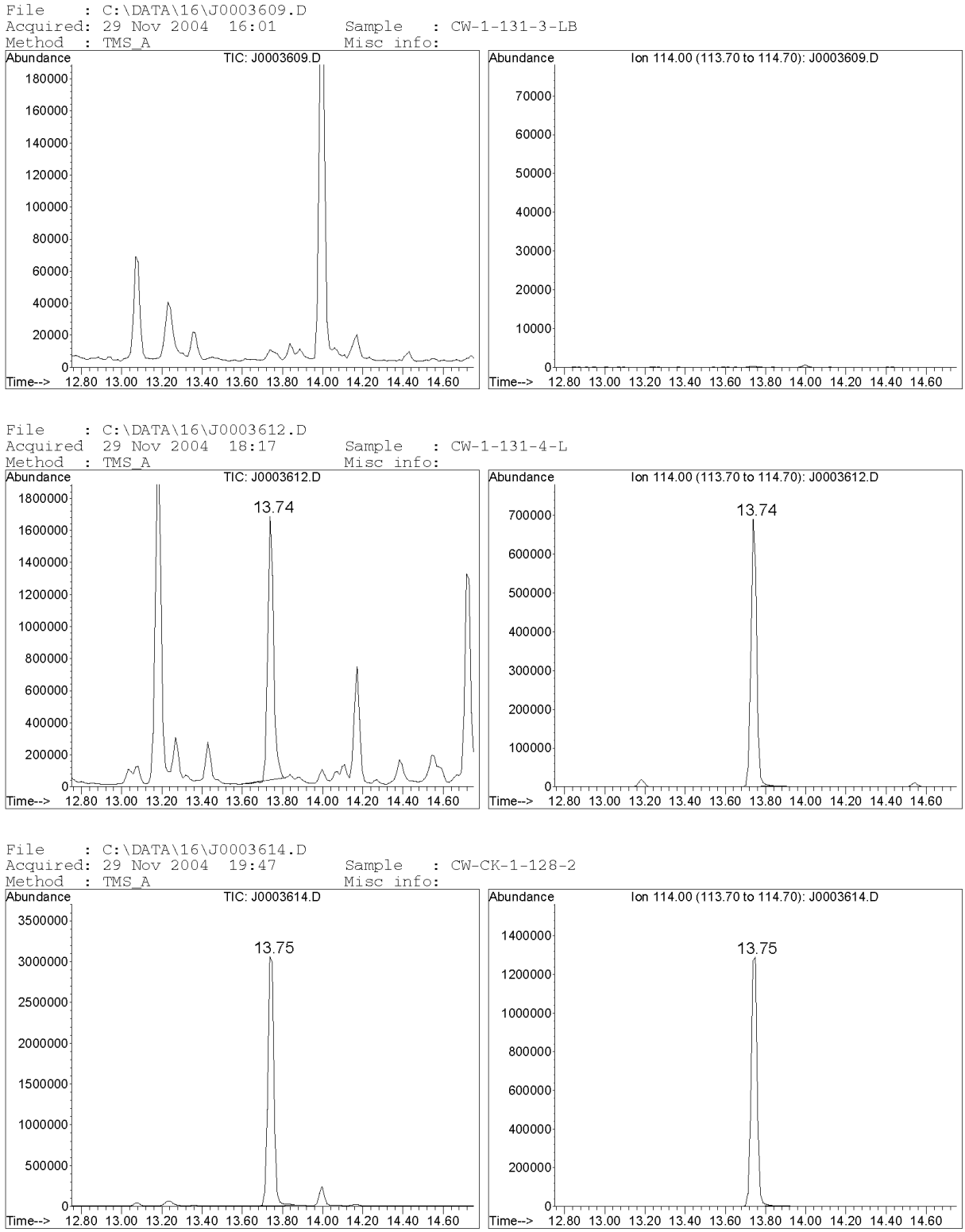
GC-EI-MS Method name:	**TMS_A**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 38 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0. 70 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	70 °C (8 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	8 min	**Scan range:**	40-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☐ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☒ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



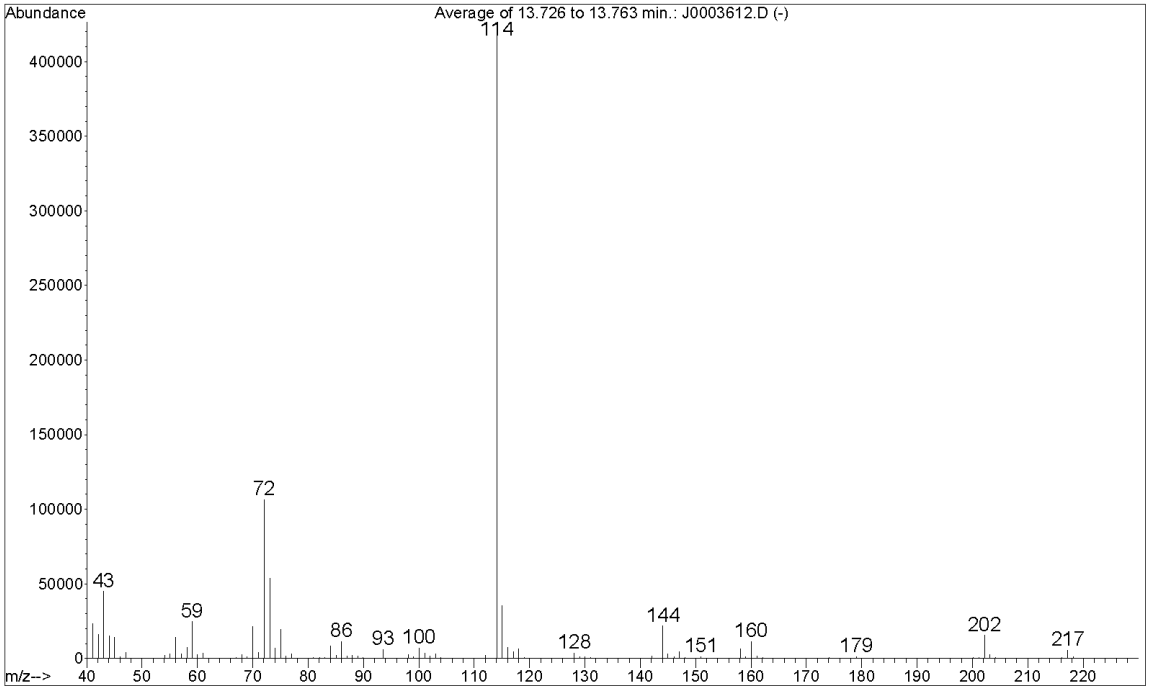
EI chromatograms supporting identification of compound **6**; TIC on left; EIC (m/z **114**) on right.

Top: Chromatograms of Liquid blank, aliquot **CW-1-131-3-LB** from **LB/31**.

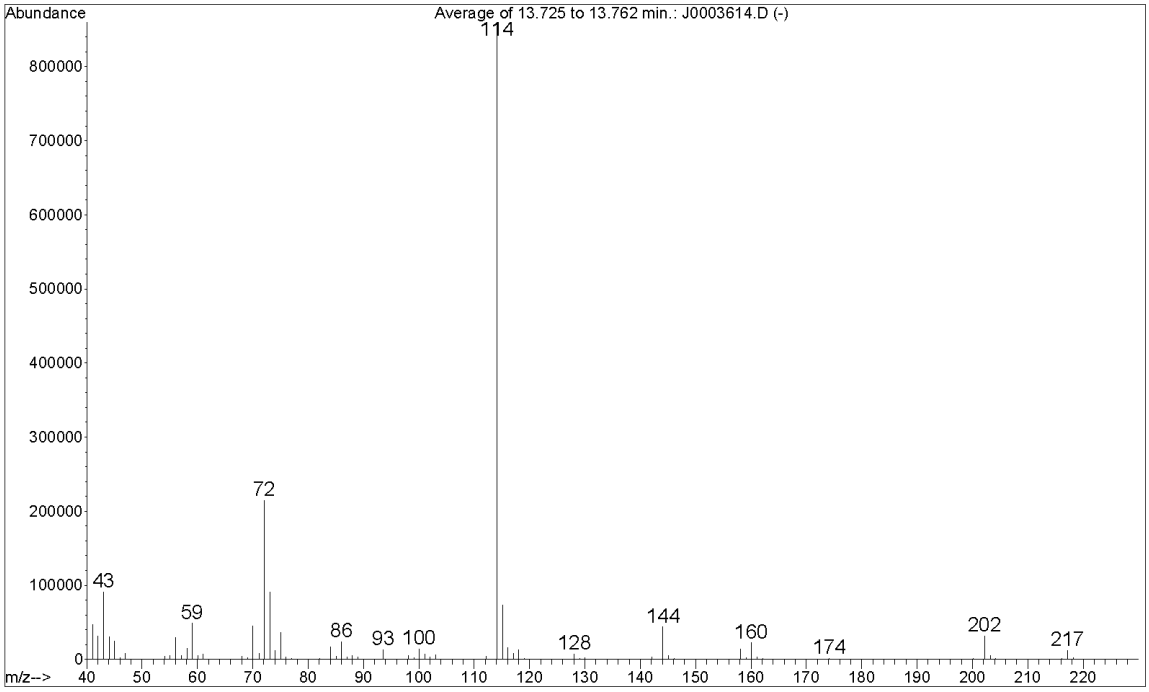
Center: Chromatograms of Liquid sample, aliquot **CW-1-131-4-L** from **L/31**, retention time **13.74** min.

Bottom: Chromatograms of TMS derivative of the standard of **2-(N,N-Diisopropylamino)ethanol** [2-(N,N-Diisopropylamino)ethyl trimethylsilyl ether], retention time **13.75** min.

```
File       : C:\DATA\16\J0003612.D
Acquired   : 29 Nov 2004  18:17     using AcqMethod TMS_A
Sample Name: CW-1-131-4-L
Misc Info  :
```




```
File       : C:\DATA\16\J0003614.D
Acquired   : 29 Nov 2004  19:47     using AcqMethod TMS_A
Sample Name: CW-CK-1-128-2
Misc Info  :
```



EI mass spectrum of:

Top: Compound **6** in Liquid sample **L/31**, aliquot **CW-1-131-4-L**

Bottom: TMS derivative of the authentic reference standard of **2-(N,N-Diisopropylamino)ethanol** [2-(N,N-Diisopropylamino)ethyl trimethylsilyl ether] corresponding to compound **6** (MW: **217**)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** L/31, LB/31 **Compound number:** 6

Aliquot codes:			
Sample:	**CW-1-131-4-L**	**Blank:**	**CW-1-131-3-LB**

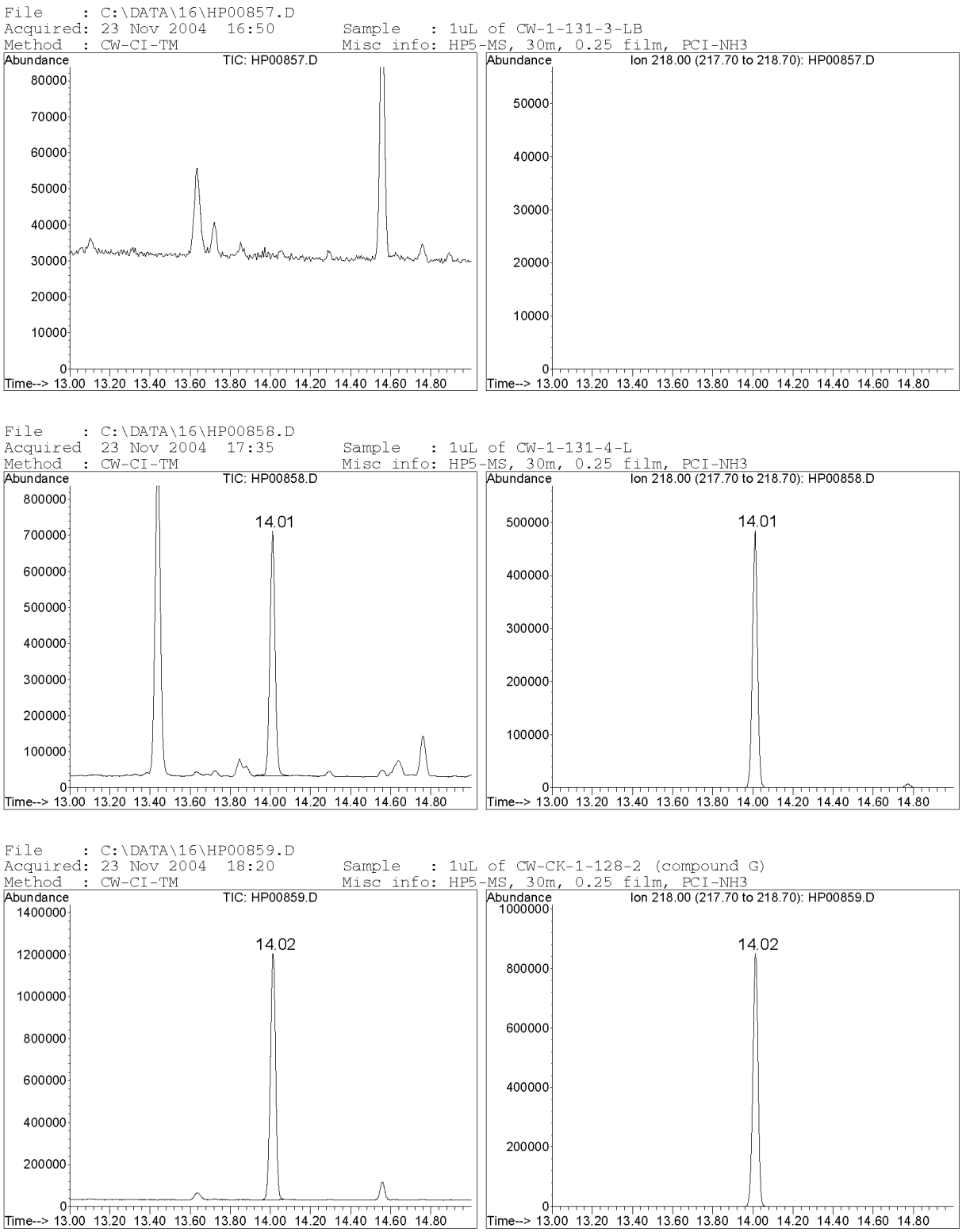
GC-CI-MS Method name:	**CW-CI-TM**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	70 °C (8 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☐ Methane ☐ Isobutane ☒ Ammonia ☐ Other:		
Solvent delay time:	8 min	**Scan range:**	50-550 m/z
Electron energy:	235 eV	**Scan time:**	0.35 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☐ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☒ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



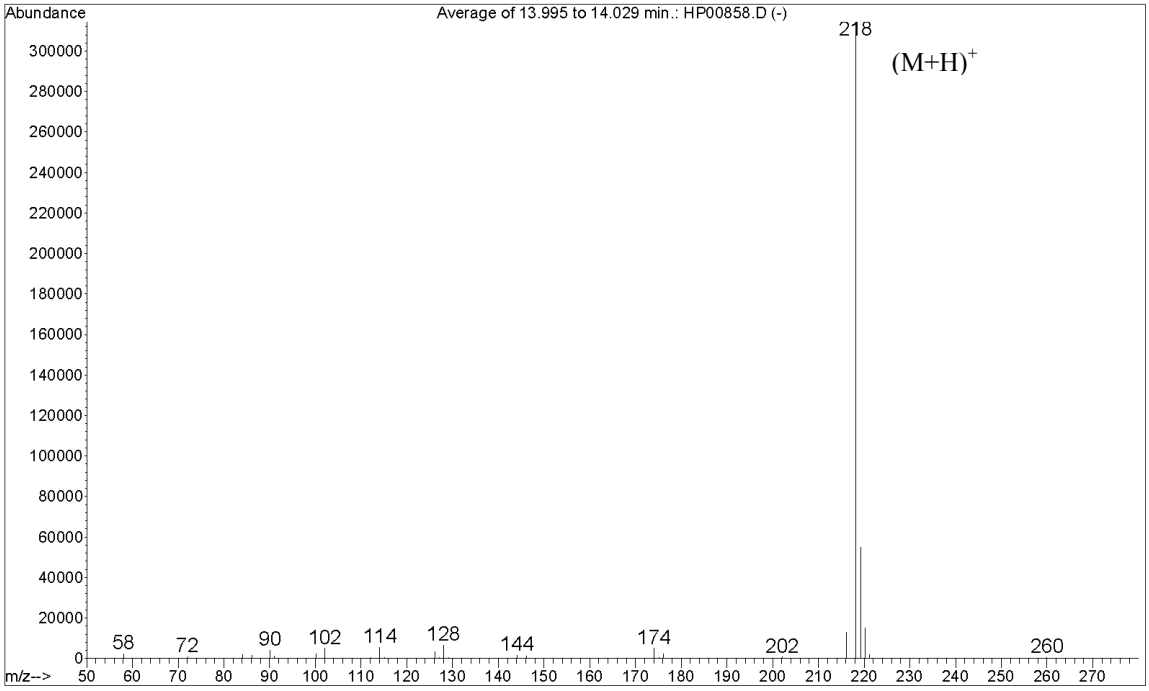
CI chromatograms supporting identification of compound **6**; TIC on left; EIC (m/z **218**) on right.

Top: Chromatograms of Liquid blank, aliquot **CW-1-131-3-LB** from **LB/31**.

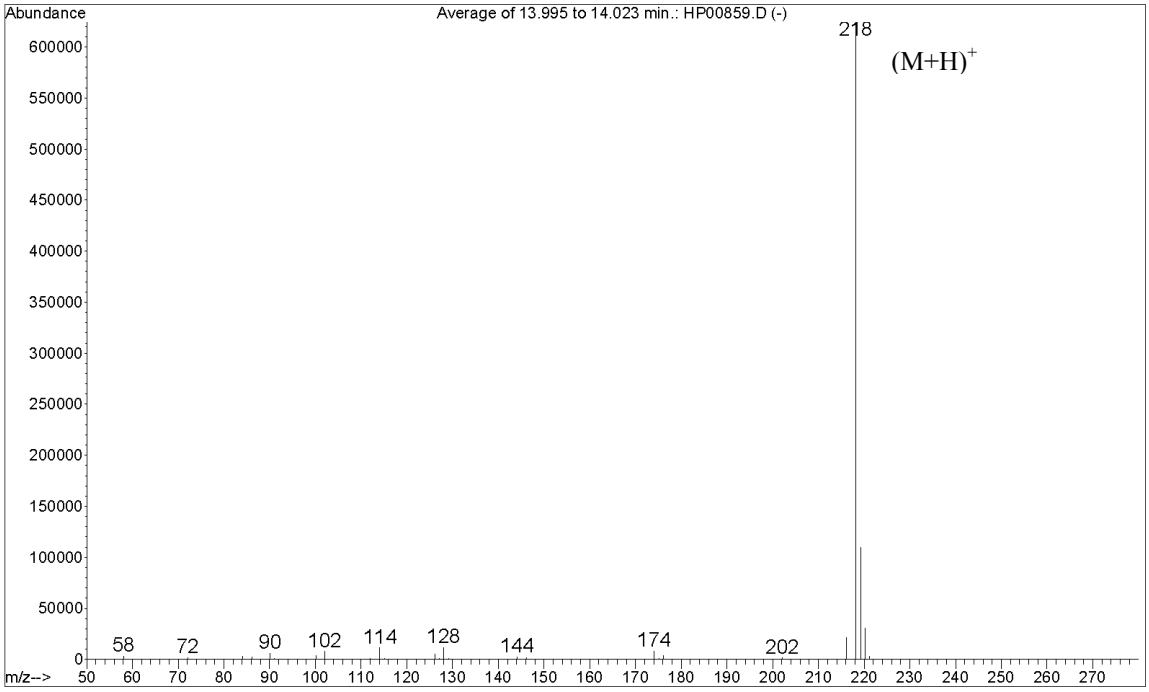
Center: Chromatograms of Liquid sample, aliquot **CW-1-131-4-L** from **L/31**, retention time **14.01** min.

Bottom: Chromatograms of TMS derivative of the standard of **2-(N,N-Diisopropylamino)ethanol** [2-(N,N-Diisopropylamino)ethyl trimethylsilyl ether], retention time **14.02** min.

```
File       : C:\DATA\16\HP00858.D
Acquired   : 23 Nov 2004  17:35     using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-1-131-4-L
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH3
```




```
File       : C:\DATA\16\HP00859.D
Acquired   : 23 Nov 2004  18:20     using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-CK-1-128-2 (compound G)
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH3
```



CI mass spectrum of:

Top: Compound **6** in Liquid sample **L/31**, aliquot **CW-1-131-4-L**

Bottom: TMS derivative of the authentic reference standard of **2-(N,N-Diisopropylamino)ethanol** [2-(N,N-Diisopropylamino)ethyl trimethylsilyl ether] corresponding to compound **6** (MW: **217**)

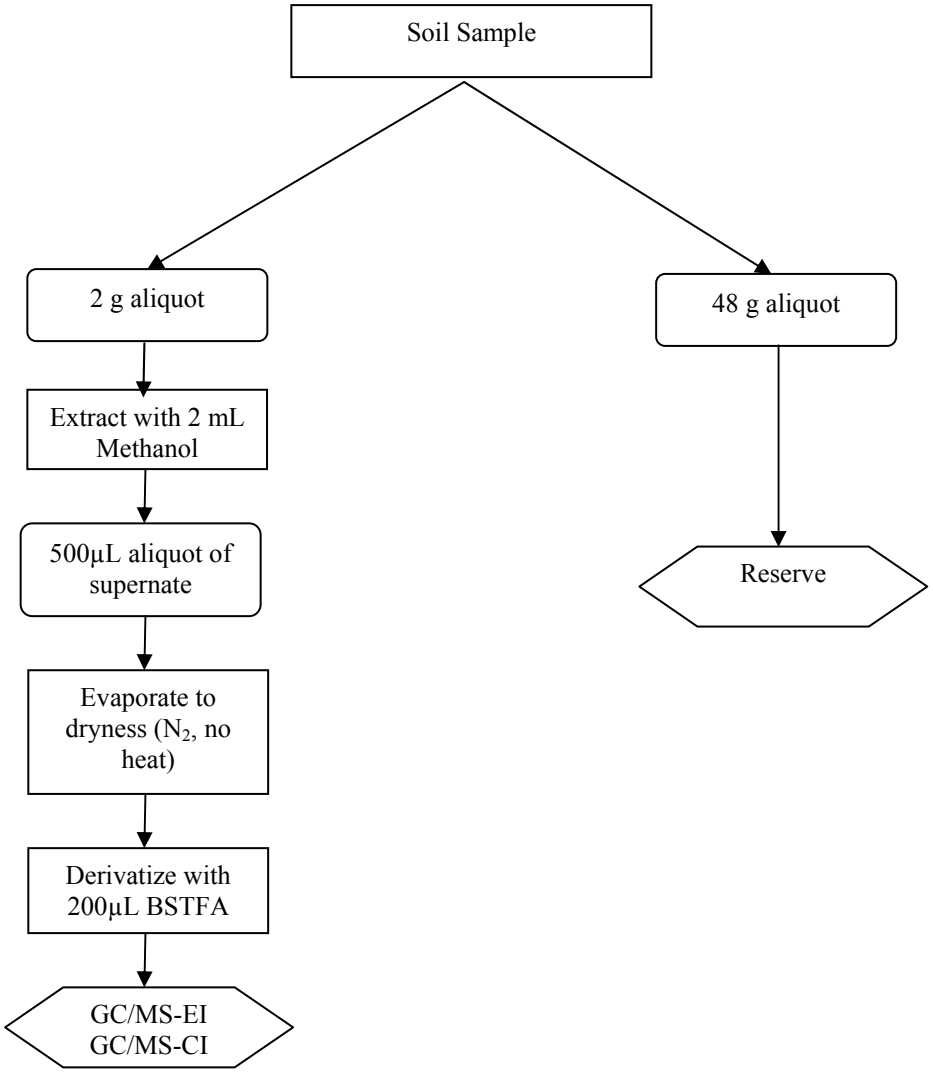
SAMPLE PREPARATION DESCRIPTION

Laboratory code: 31 **Sample code(s):** S/31 **Sample Blank code:** SB/31

1. Sample preparation

Sample/ Aliquot Code	*Specification of Sample/ Type of Sample Preparation*	**Amount/ Volume**	*Sample Preparation Procedures*	**End Volume**	**Resulting Aliquot Code**	**Analytical Technique(s)**
S/31 SB/31	TMS derivative of methanol extract of sample	2 g	2 mL Methanol was added to 2 g of soil and sonicated for 15 minutes followed by 4 minutes of centrifuge. 500µL of the supernate was dried (N2, no heat). 200 µL BSTFA was added and heated at 60°C for 30 minutes.	200 µL	CW-1-131-2-S CW-1-131-1-SB	GC/MS-EI GC/MS-CI

2. Additional information



Note: This flowchart is for visualization only; see the preceding sample preparation description page for sample aliquot numbers

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** S/31, SB/31 **Compound number:** 7

Aliquot codes:			
Sample:	**CW-1-131-2-S**	**Blank:**	**CW-1-131-1-SB**

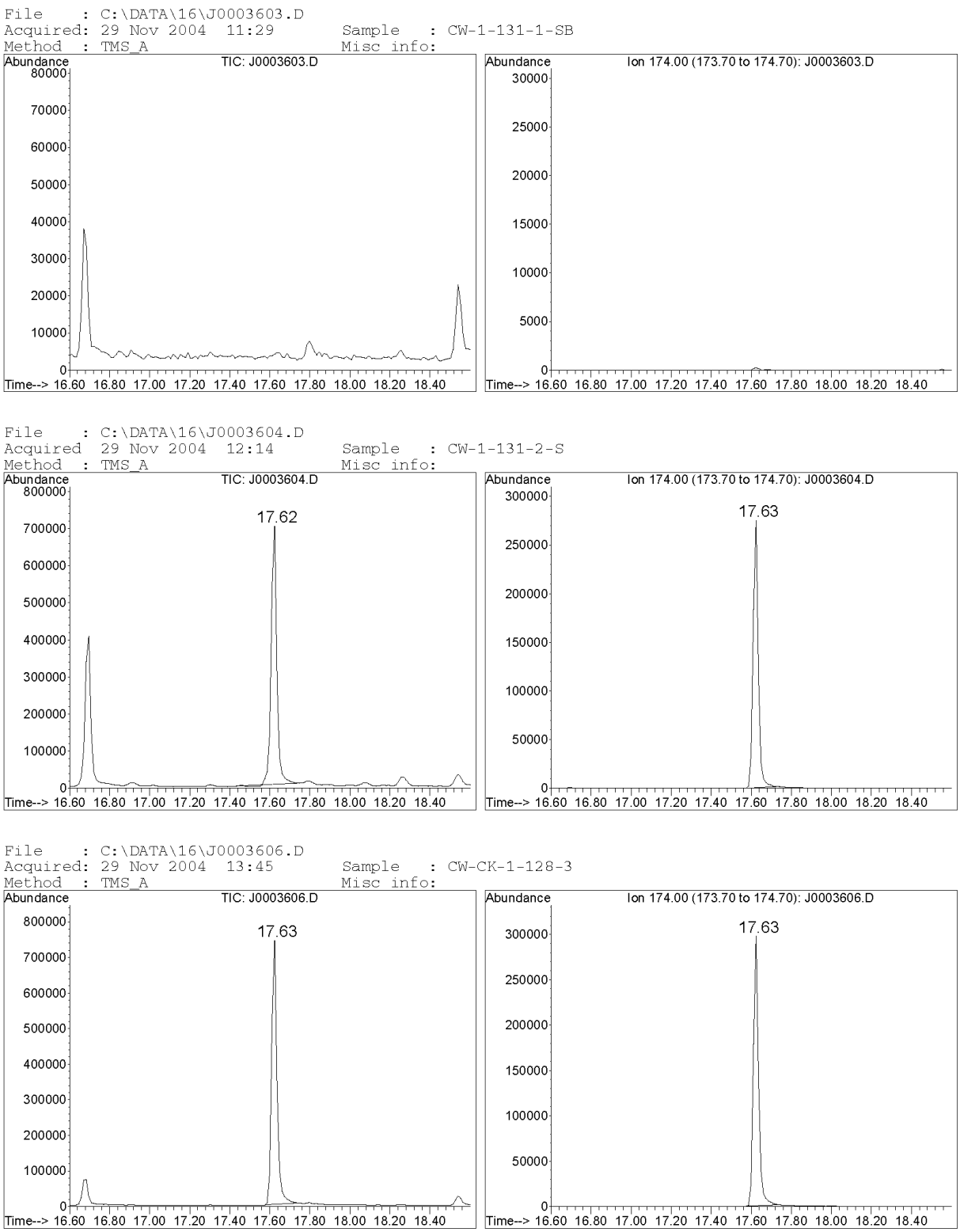
GC-EI-MS Method name:	**TMS_A**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 38 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0. 70 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	70 °C (8 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	8 min	**Scan range:**	40-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☐ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☒ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



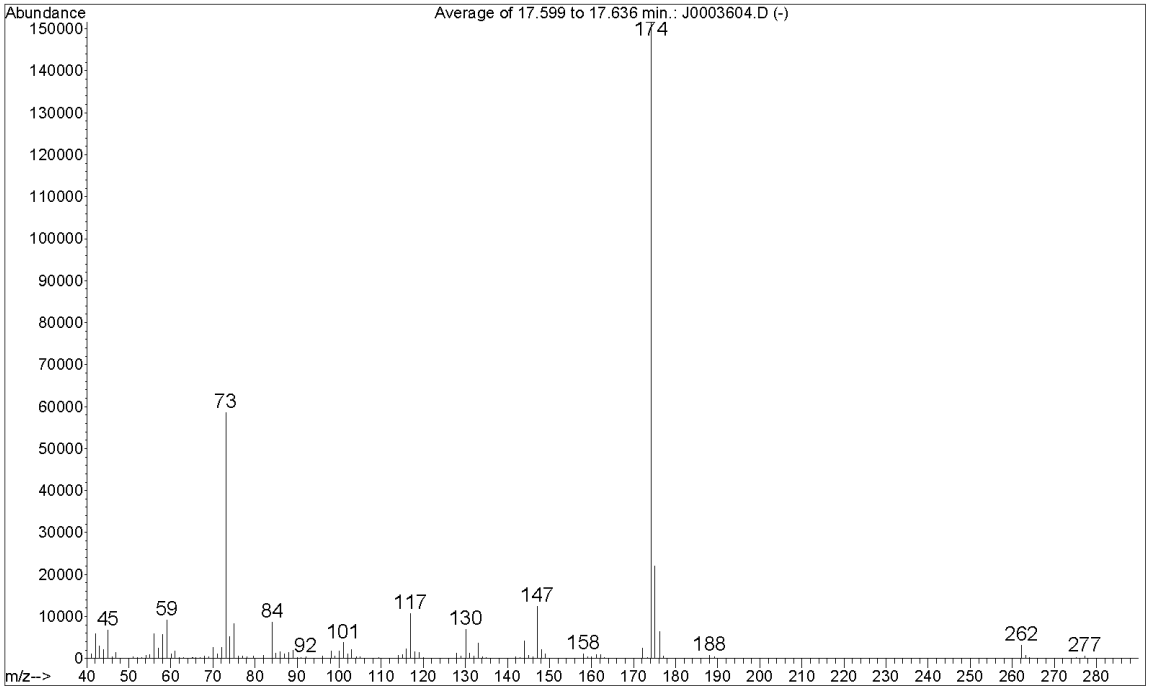
EI chromatograms supporting identification of compound **7**; TIC on left; EIC (m/z **174**) on right.

Top: Chromatograms of Soil blank, aliquot **CW-1-131-1-SB** from **SB/31**.

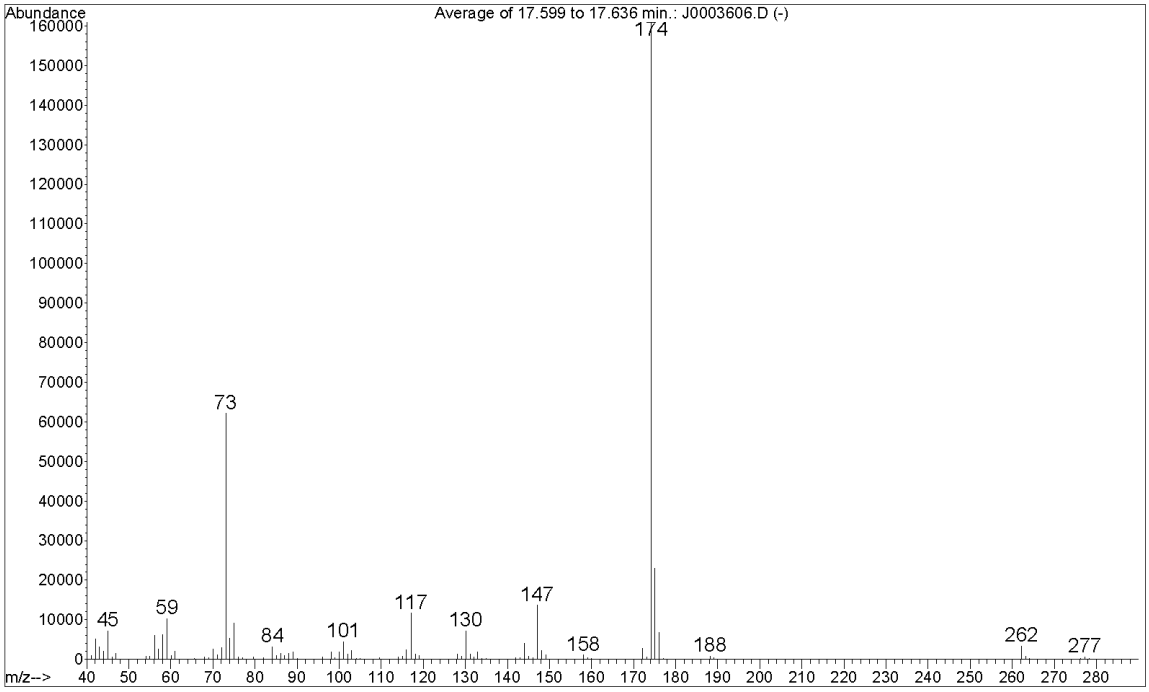
Center: Chromatograms of Soil sample, aliquot **CW-1-131-2-S** from **S/31**, retention time **17.62** min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Ethyldiethanolamine** [Bis(2-trimethylsilyloxyethyl)ethylamine], retention time **17.63** min.

```
File       : C:\DATA\16\J0003604.D
Acquired   : 29 Nov 2004  12:14     using AcqMethod TMS_A
Sample Name: CW-1-131-2-S
Misc Info  :
```




```
File       : C:\DATA\16\J0003606.D
Acquired   : 29 Nov 2004  13:45     using AcqMethod TMS_A
Sample Name: CW-CK-1-128-3
Misc Info  :
```



EI mass spectrum of:

Top: Compound **7** in Soil sample **S/31**, aliquot **CW-1-131-2-S**

Bottom: TMS derivative of the authentic reference standard of **Ethyldiethanolamine** [Bis(2-trimethylsilyloxyethyl)ethylamine] corresponding to compound **7** (MW: **277**)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** S/31, SB/31 **Compound number:** 7

Aliquot codes:			
Sample:	**CW-1-131-2-S**	**Blank:**	**CW-1-131-1-SB**

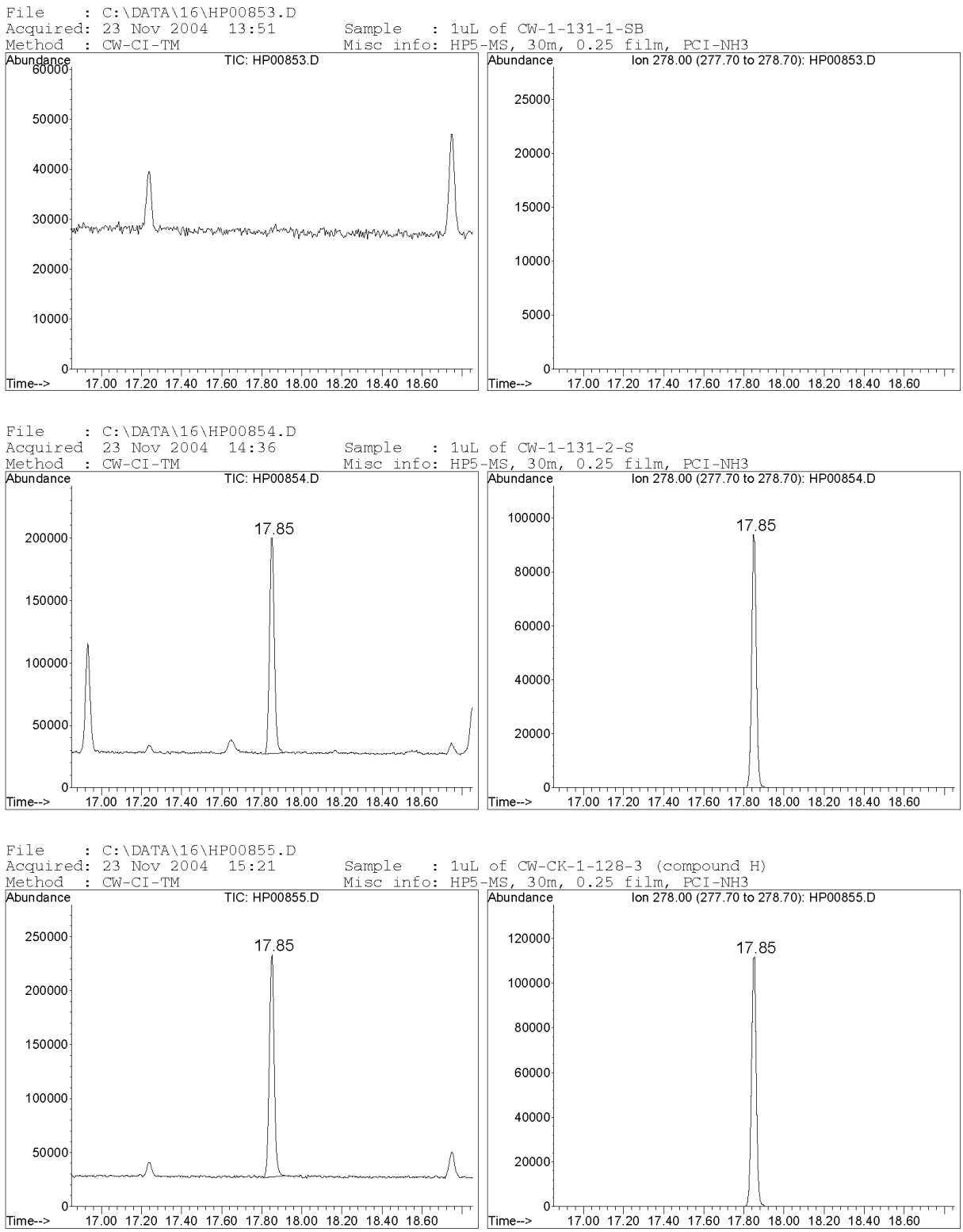
GC-CI-MS Method name:	**CW-CI-TM**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	70 °C (8 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☐ Methane ☐ Isobutane ☒ Ammonia ☐ Other:		
Solvent delay time:	8 min	**Scan range:**	50-550 m/z
Electron energy:	235 eV	**Scan time:**	0.35 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☐ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☒ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



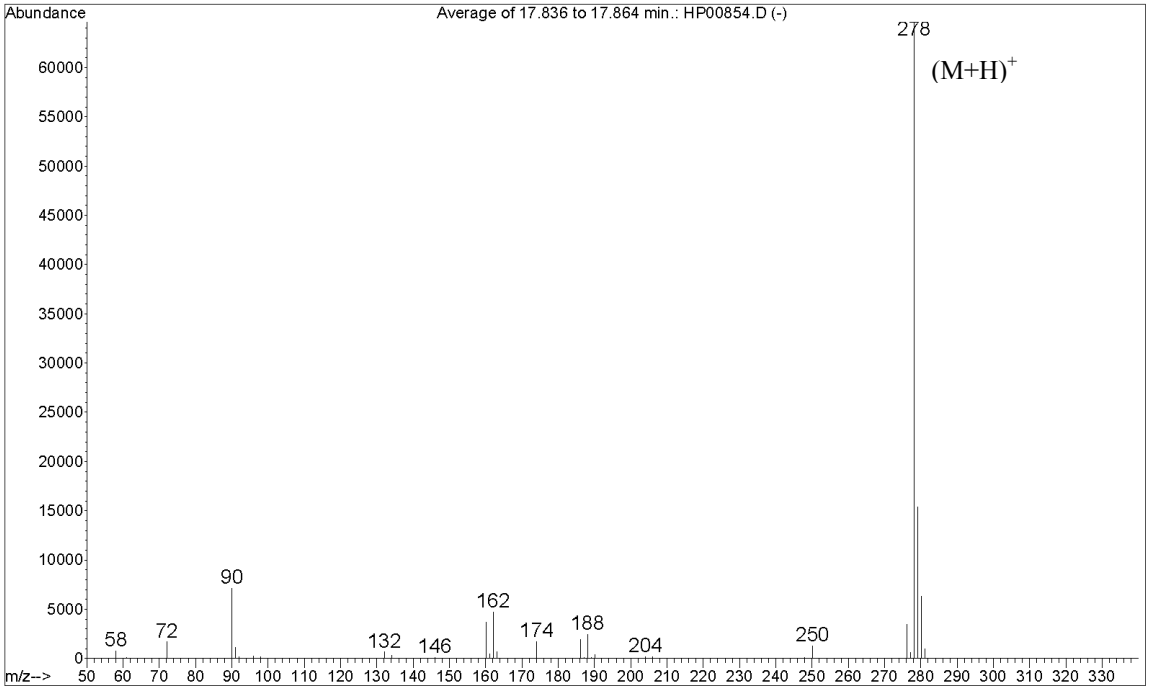
CI chromatograms supporting identification of compound **7**; TIC on left; EIC (m/z **278**) on right.

Top: Chromatograms of Soil blank, aliquot **CW-1-131-1-SB** from **SB/31**.

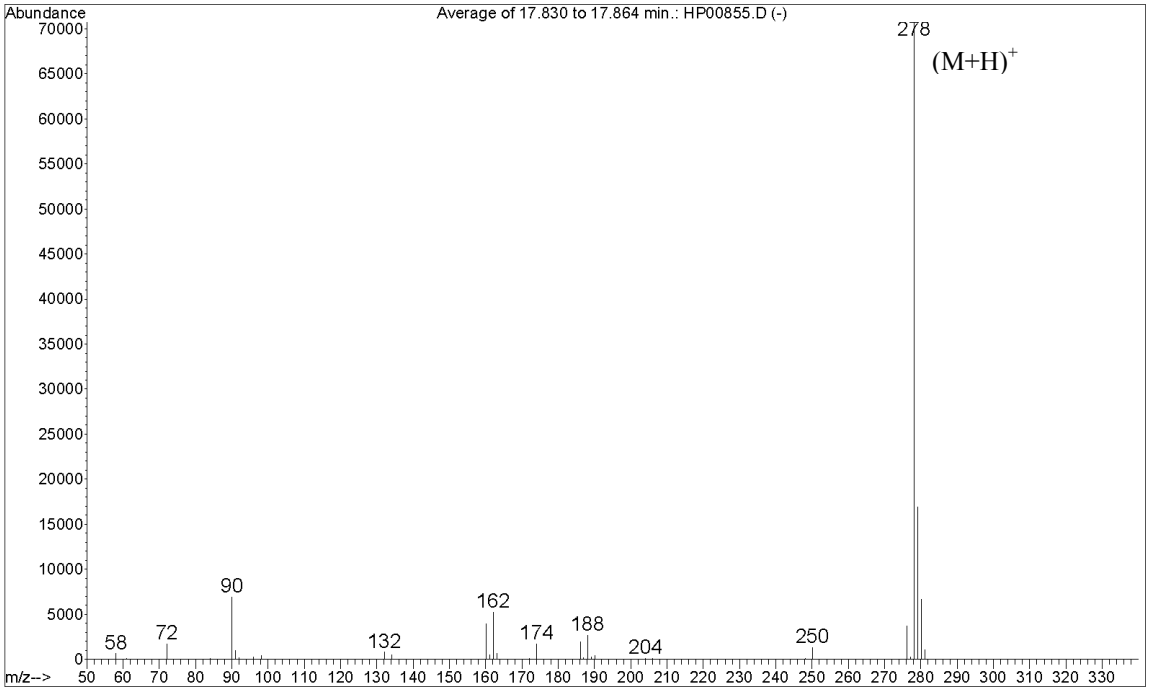
Center: Chromatograms of Soil sample, aliquot **CW-1-131-2-S** from **S/31**, retention time **17.85** min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Ethyldiethanolamine** [Bis(2-trimethylsilyloxyethyl)ethylamine], retention time **17.85** min.

```
File       : C:\DATA\16\HP00854.D
Acquired   : 23 Nov 2004  14:36     using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-1-131-2-S
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH3
```




```
File       : C:\DATA\16\HP00855.D
Acquired   : 23 Nov 2004  15:21     using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-CK-1-128-3 (compound H)
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH3
```



CI mass spectrum of:

Top: Compound **7** in Soil sample **S/31**, aliquot **CW-1-131-2-S**

Bottom: TMS derivative of the authentic reference standard of **Ethyldiethanolamine** [Bis(2-trimethylsilyloxyethyl)ethylamine]corresponding to compound **7** (MW: **277**)

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** S/31, SB/31 **Compound number:** 8

Aliquot codes:			
Sample:	**CW-1-131-2-S**	**Blank:**	**CW-1-131-1-SB**

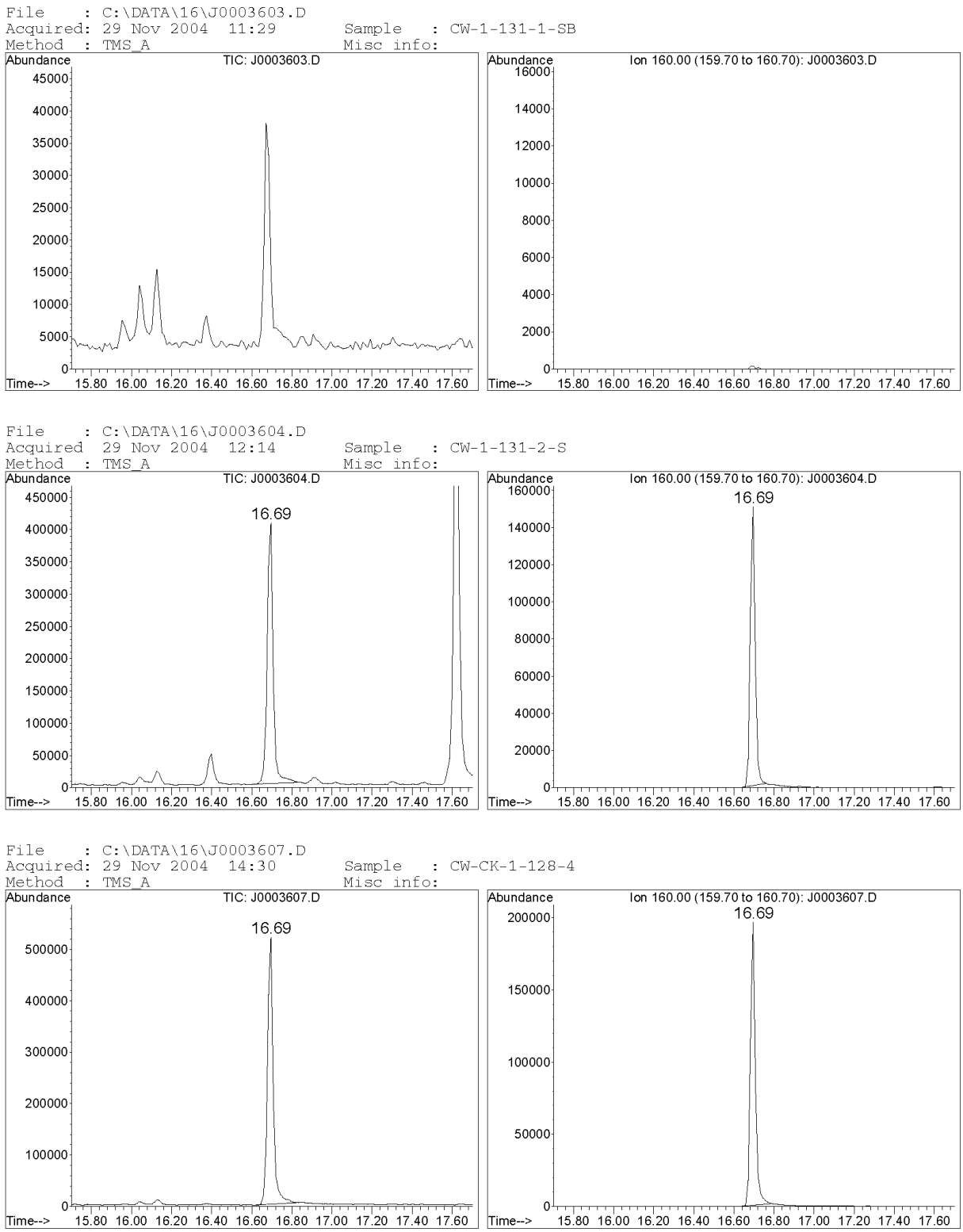
GC-EI-MS Method name:	**TMS_A**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 38 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0. 70 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	70 °C (8 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	8 min	**Scan range:**	40-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☐ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☒ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



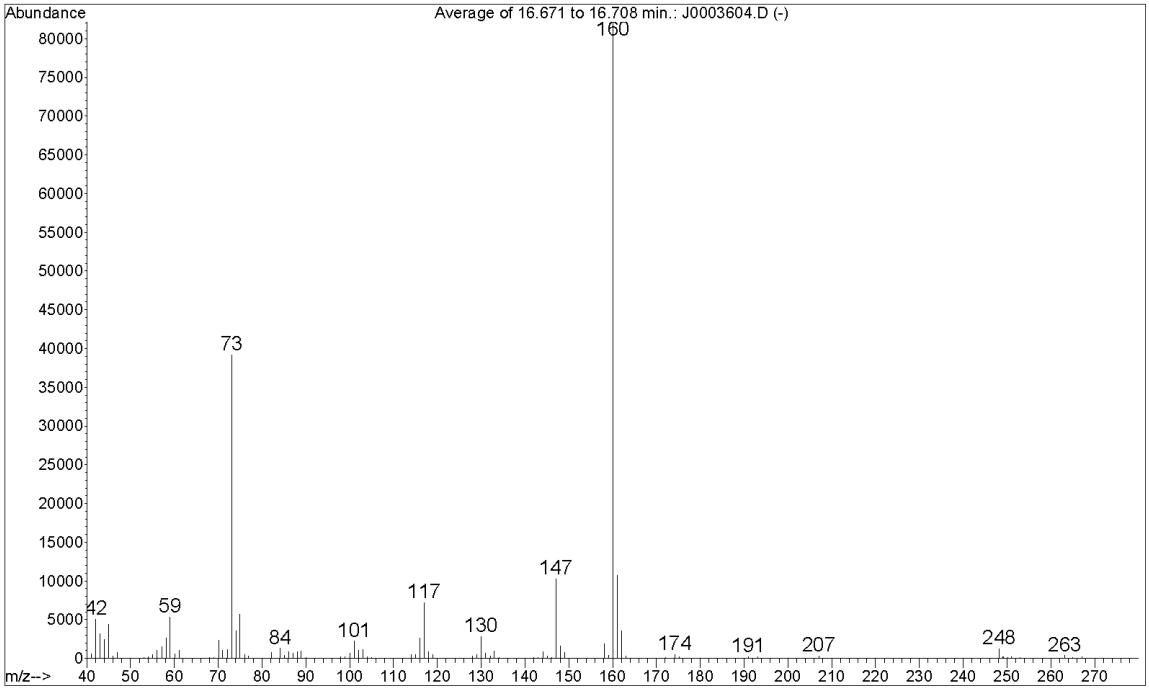
EI chromatograms supporting identification of compound **8**; TIC on left; EIC (m/z **160**) on right.

Top: Chromatograms of Soil blank, aliquot **CW-1-131-1-SB** from **SB/31**.

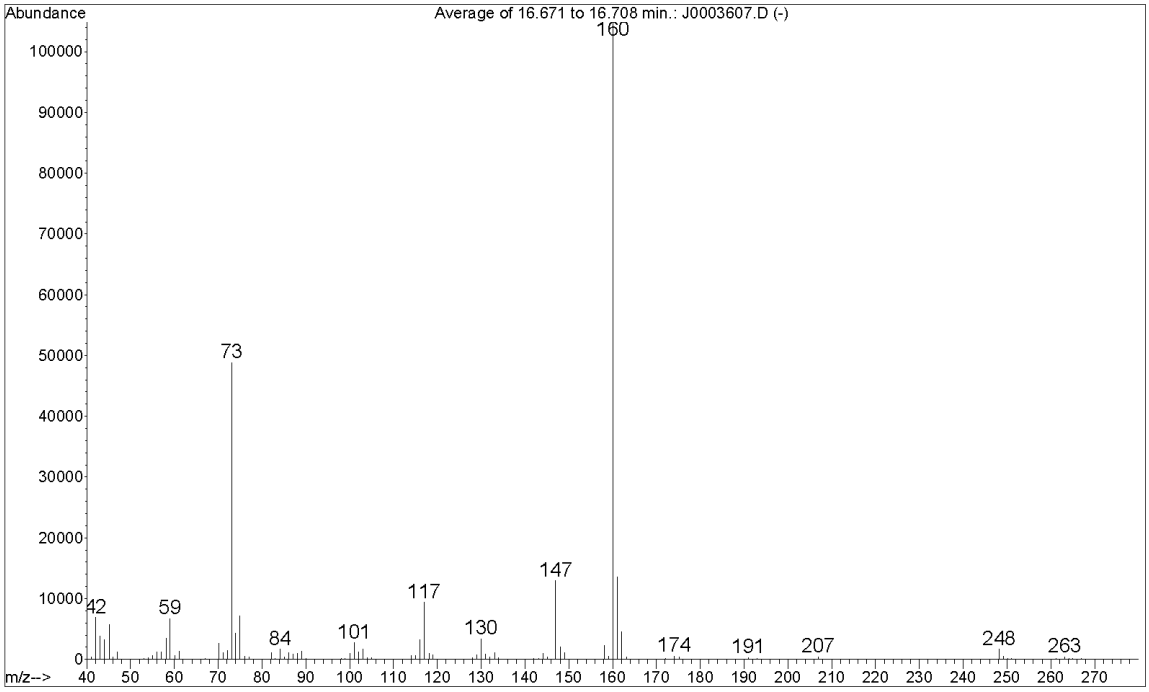
Center: Chromatograms of Soil sample, aliquot **CW-1-131-2-S** from **S/31**, retention time **16.69** min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Methyldiethanolamine** [Bis(2-trimethylsilyloxyethyl)methylamine], retention time **16.69** min.

```
File       : C:\DATA\16\J0003604.D
Acquired   : 29 Nov 2004  12:14     using AcqMethod TMS_A
Sample Name: CW-1-131-2-S
Misc Info  :
```




```
File       : C:\DATA\16\J0003607.D
Acquired   : 29 Nov 2004  14:30     using AcqMethod TMS_A
Sample Name: CW-CK-1-128-4
Misc Info  :
```



EI mass spectrum of:

Top: Compound **8** in Soil sample **S/31**, aliquot **CW-1-131-2-S**

Bottom: TMS derivative of the authentic reference standard of **Methyldiethanolamine** [Bis(2-trimethylsilyloxyethyl)methylamine] corresponding to compound **8** (MW: **263**)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** S/31, SB/31 **Compound number:** 8

Aliquot codes:			
Sample:	**CW-1-131-2-S**	**Blank:**	**CW-1-131-1-SB**

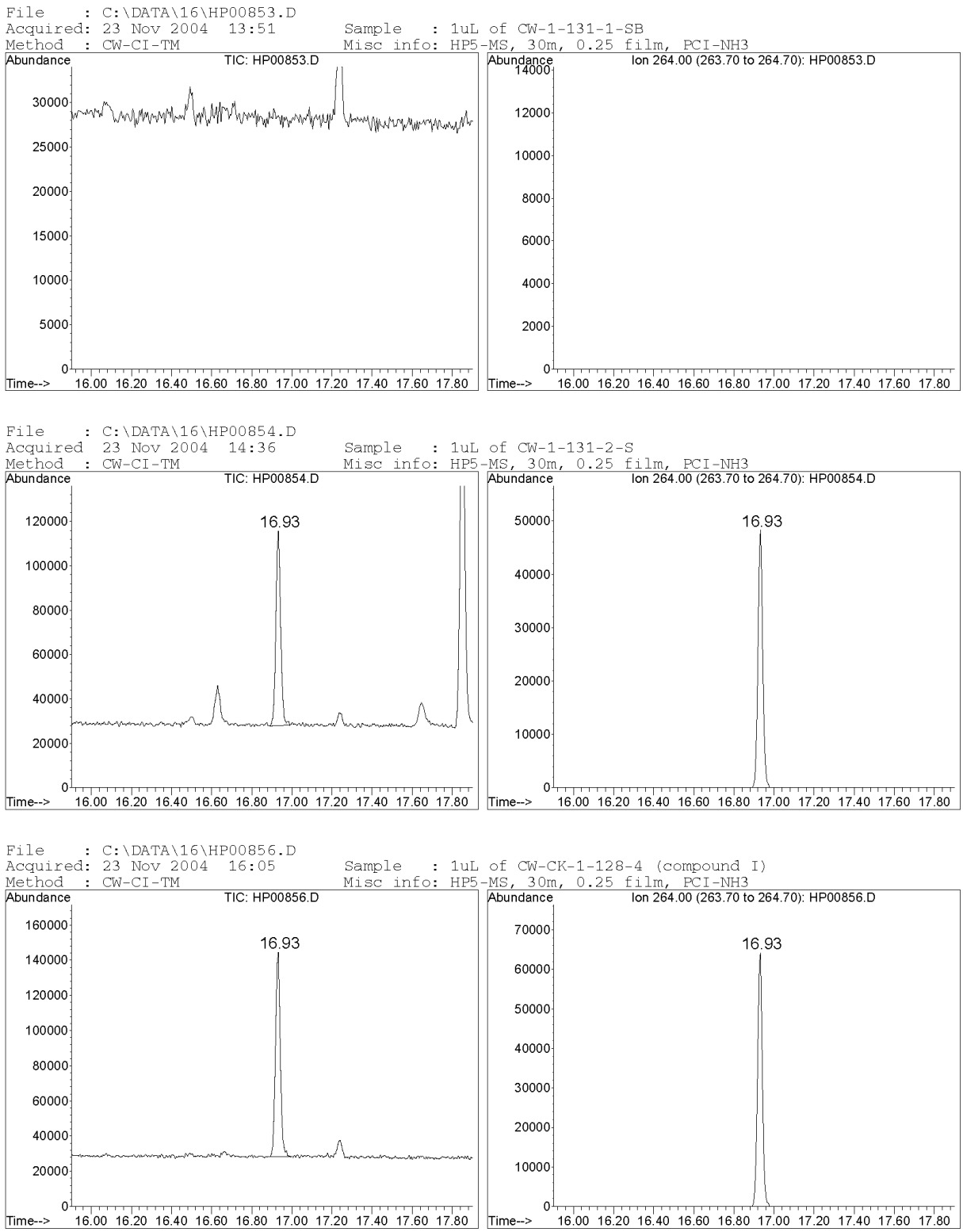
GC-CI-MS Method name:	**CW-CI-TM**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	70 °C (8 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☐ Methane ☐ Isobutane ☒ Ammonia ☐ Other:		
Solvent delay time:	8 min	**Scan range:**	50-550 m/z
Electron energy:	235 eV	**Scan time:**	0.35 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☐ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☒ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



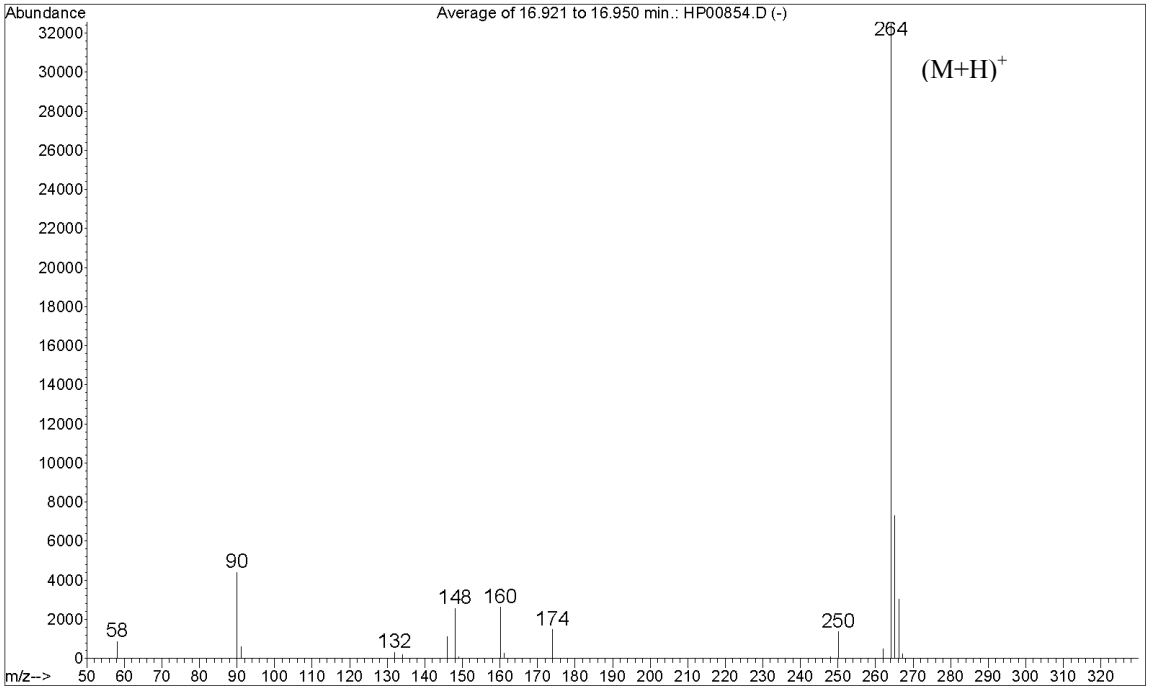
CI chromatograms supporting identification of compound **8**; TIC on left; EIC (m/z **264**) on right.

Top: Chromatograms of Soil blank, aliquot **CW-1-131-1-SB** from **SB/31**.

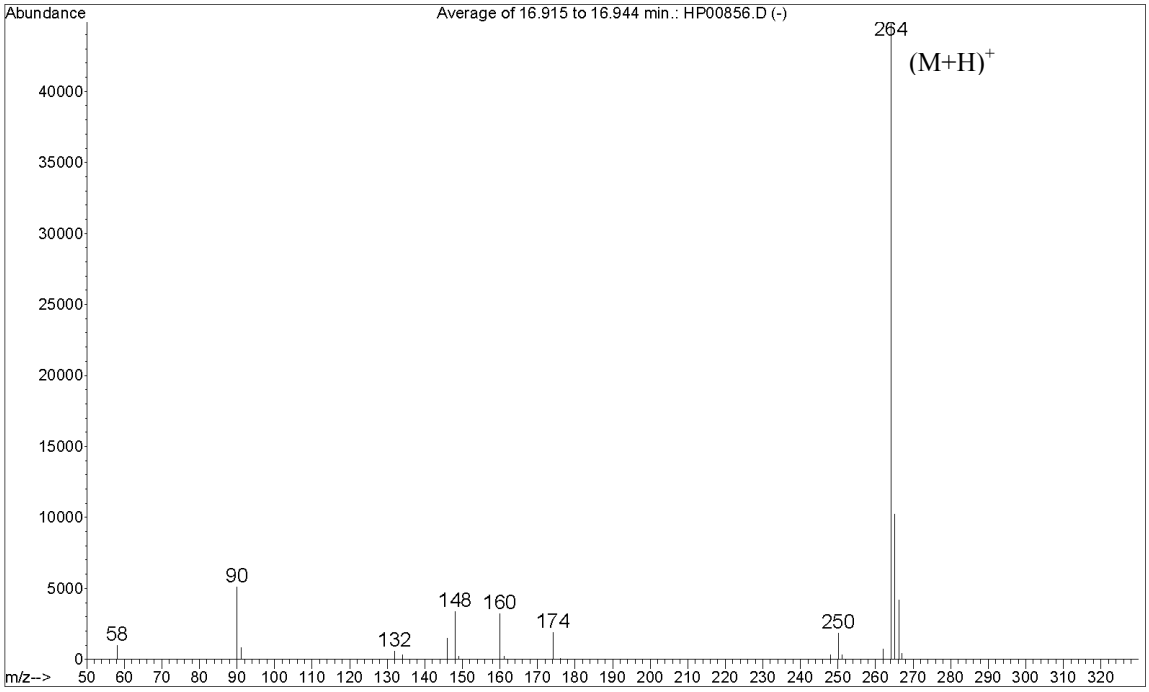
Center: Chromatograms of Soil sample, aliquot **CW-1-131-2-S** from **S/31**, retention time **16.93** min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Methyldiethanolamine** [Bis(2-trimethylsilyloxyethyl)methylamine], retention time **16.93** min.

```
File       : C:\DATA\16\HP00854.D
Acquired   : 23 Nov 2004  14:36     using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-1-131-2-S
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH3
```




```
File       : C:\DATA\16\HP00856.D
Acquired   : 23 Nov 2004  16:05     using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-CK-1-128-4 (compound I)
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH3
```



CI mass spectrum of:

Top: Compound **8** in Soil sample **S/31**, aliquot **CW-1-131-2-S**

Bottom: TMS derivative of the authentic reference standard of **Methyldiethanolamine** [Bis(2-trimethylsilyloxyethyl)methylamine] corresponding to compound **8** (MW: **263**)

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** S/31, SB/31 **Compound number:** 9

Aliquot codes:			
Sample:	**CW-1-131-2-S**	**Blank:**	**CW-1-131-1-SB**

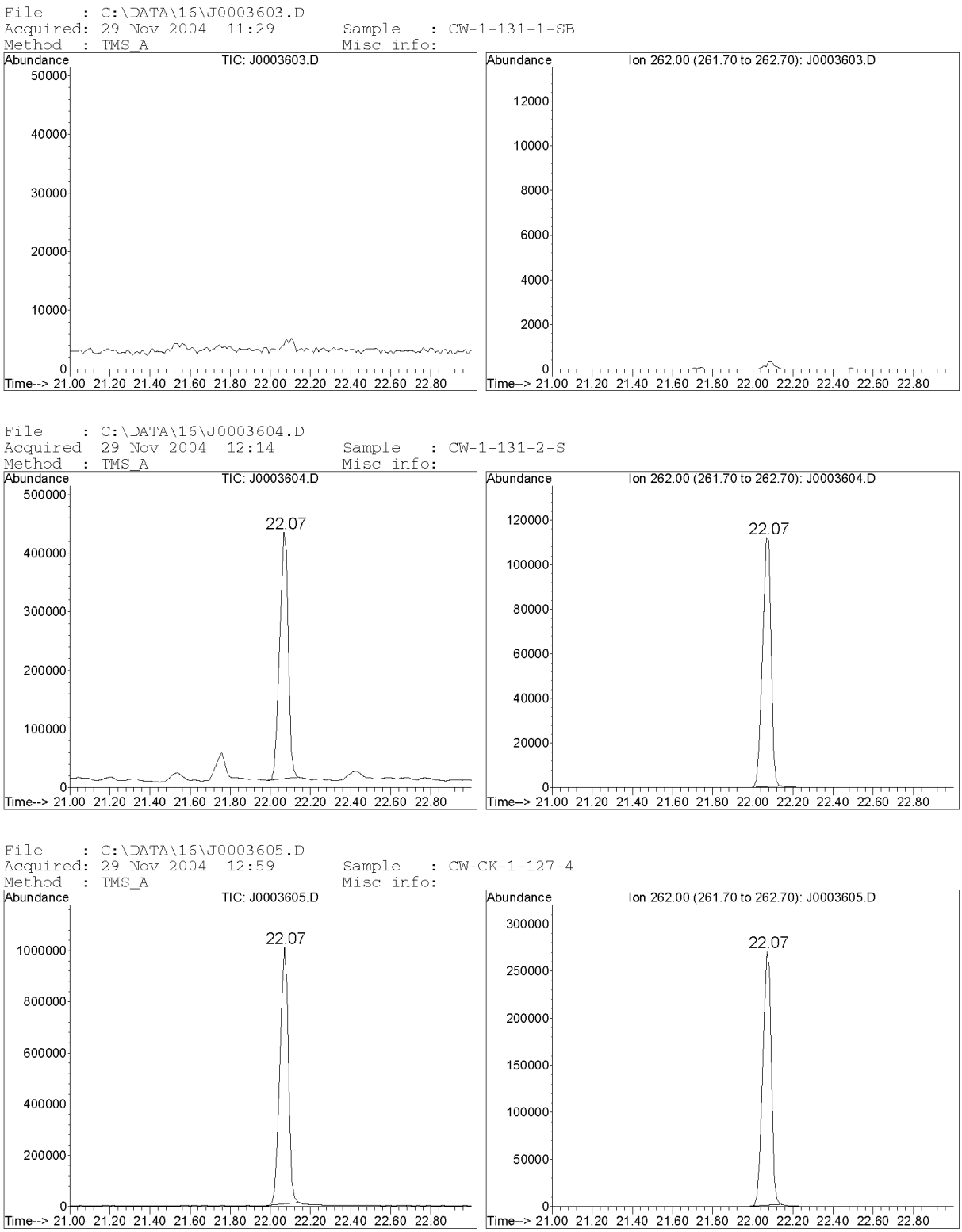
GC-EI-MS Method name:	**TMS_A**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 38 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.70 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	70 °C (8 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	8 min	**Scan range:**	40-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☐ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☒ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



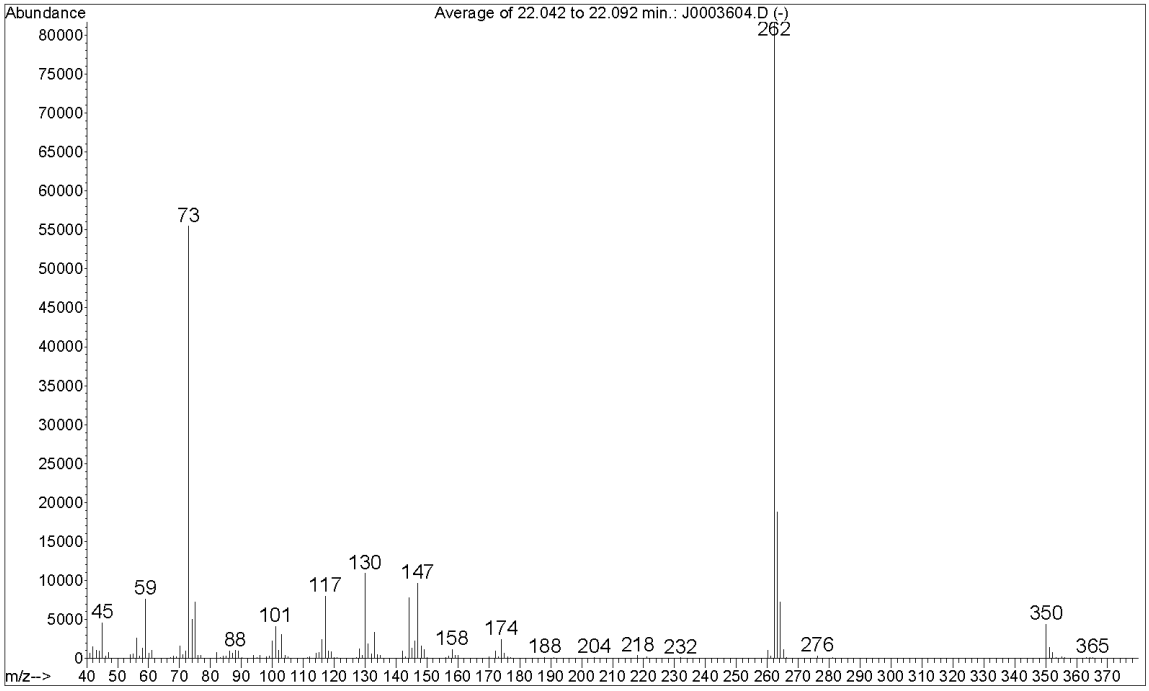
EI chromatograms supporting identification of compound **9**; TIC on left; EIC (m/z **262**) on right.

Top: Chromatograms of Soil blank, aliquot **CW-1-131-1-SB** from **SB/31**.

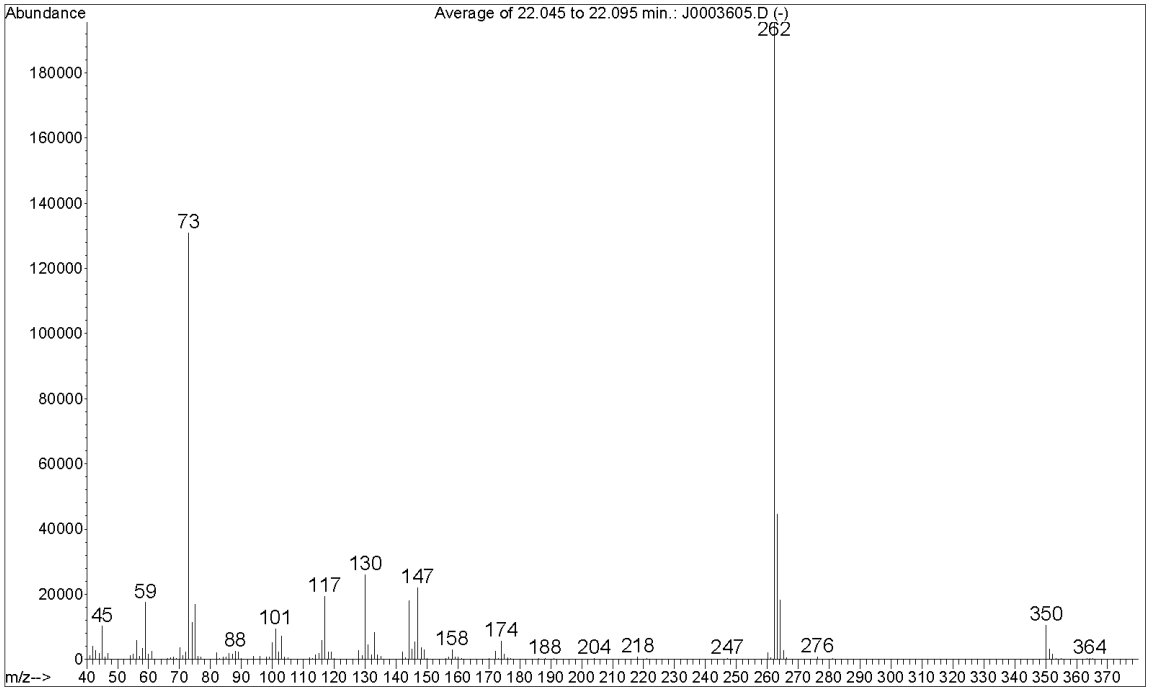
Center: Chromatograms of Soil sample, aliquot **CW-1-131-2-S** from **S/31**, retention time **22.07** min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Triethanolamine** [tris(2-trimethylsiloxyethyl)amine], retention time **22.07** min.

```
File       : C:\DATA\16\J0003604.D
Acquired   : 29 Nov 2004  12:14     using AcqMethod TMS_A
Sample Name: CW-1-131-2-S
Misc Info  :
```




```
File       : C:\DATA\16\J0003605.D
Acquired   : 29 Nov 2004  12:59     using AcqMethod TMS_A
Sample Name: CW-CK-1-127-4
Misc Info  :
```



EI mass spectrum of:

Top: Compound **9** in Soil sample **S/31**, aliquot **CW-1-131-2-S**

Bottom: TMS derivative of the authentic reference standard of **Triethanolamine** [tris(2-trimethylsiloxyethyl)amine] corresponding to compound **9** (MW: **365**)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 **Sample code(s):** S/31, SB/31 **Compound number:** 9

Aliquot codes:			
Sample:	**CW-1-131-2-S**	**Blank:**	**CW-1-131-1-SB**

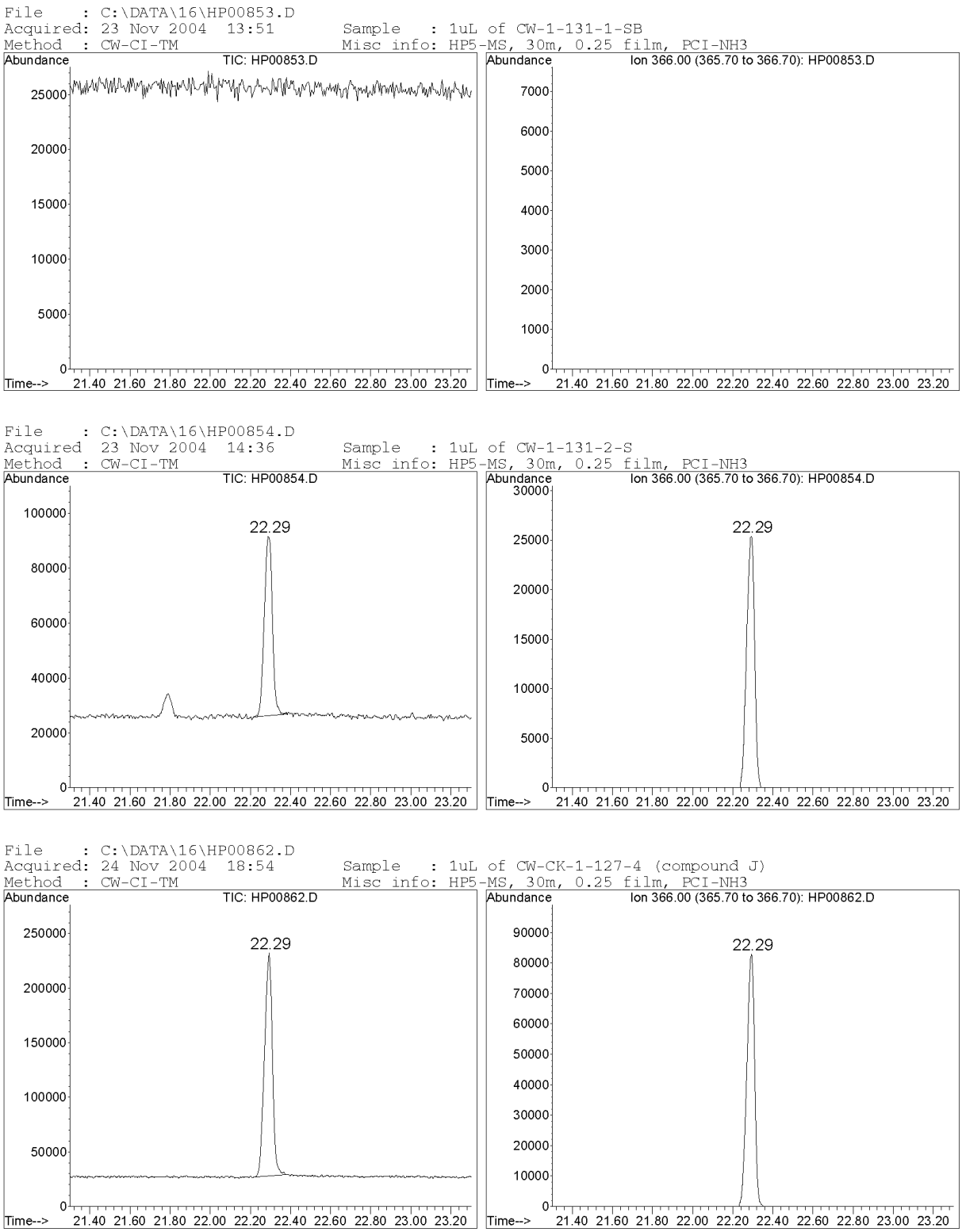
GC-CI-MS Method name:	**CW-CI-TM**

METHOD DESCRIPTION

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column phase:	5% diphenyl 95% dimethyl polysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	70 °C (8 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☐ Methane ☐ Isobutane ☒ Ammonia ☐ Other:		
Solvent delay time:	8 min	**Scan range:**	50-550 m/z
Electron energy:	235 eV	**Scan time:**	0.35 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

ANALYSIS

Compound identified as:	☐ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☒ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



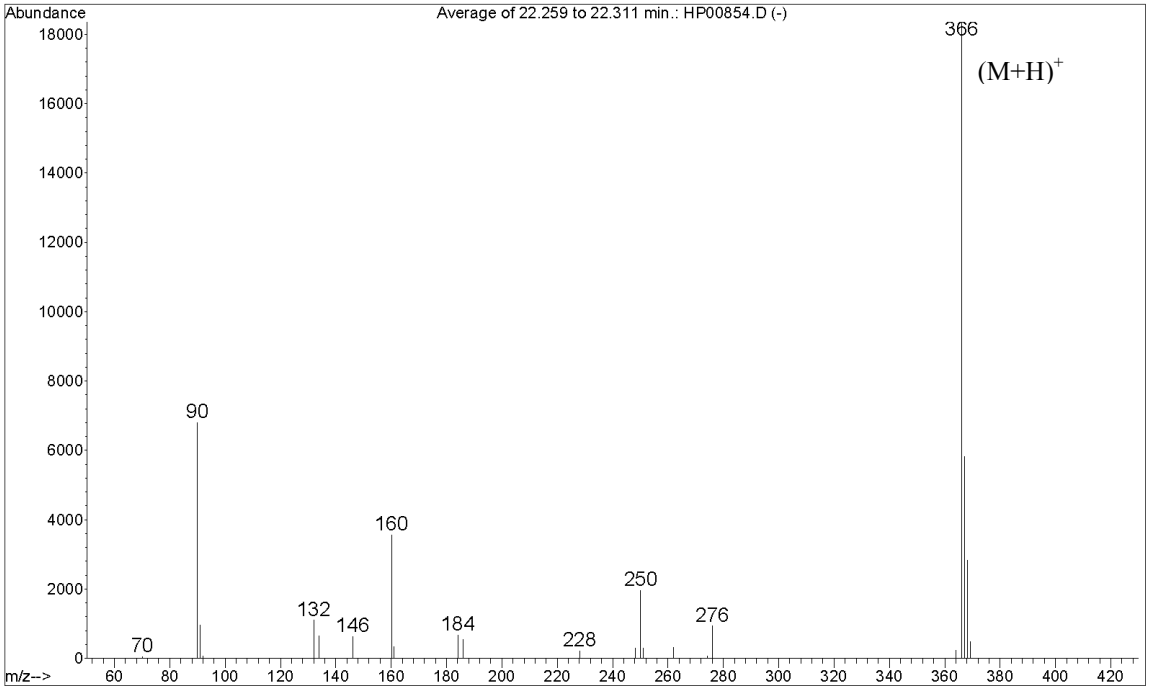
CI chromatograms supporting identification of compound **9**; TIC on left; EIC (m/z **366**) on right.

Top: Chromatograms of Soil blank, aliquot **CW-1-131-1-SB** from **SB/31**.

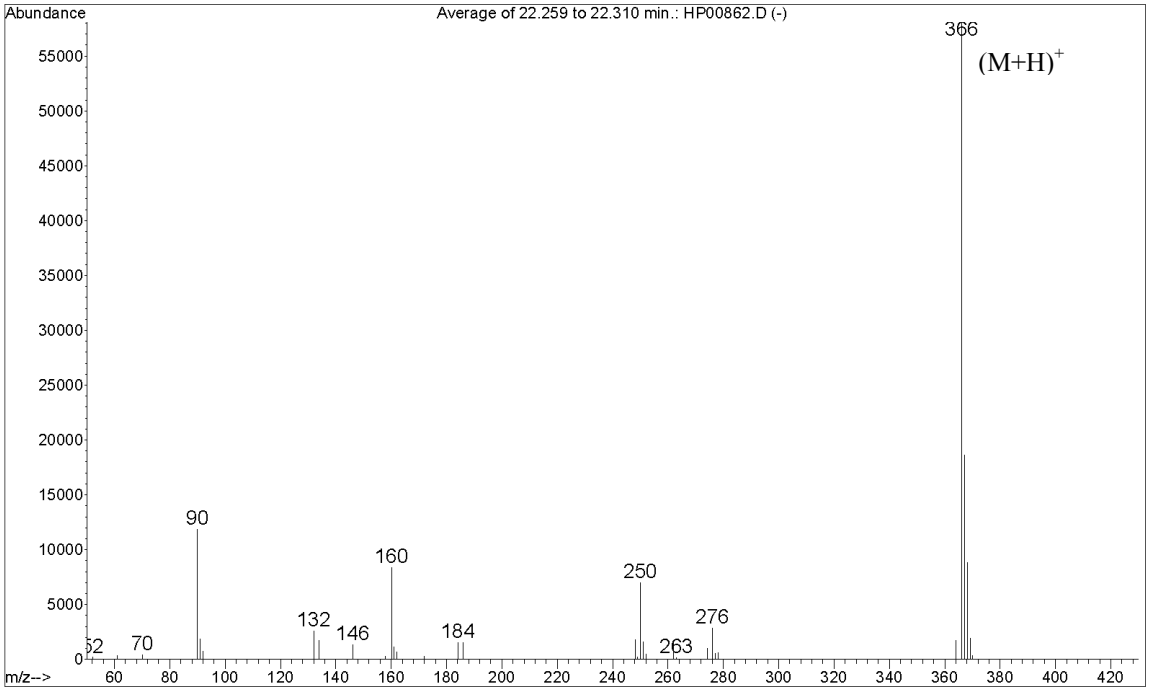
Center: Chromatograms of Soil sample, aliquot **CW-1-131-2-S** from **S/31**, retention time **22.29** min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Triethanolamine** [tris(2-trimethylsiloxyethyl)amine], retention time **22.29** min.

```
File       : C:\DATA\16\HP00854.D
Acquired   : 23 Nov 2004  14:36     using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-1-131-2-S
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH3
```




```
File       : C:\DATA\16\HP00862.D
Acquired   : 24 Nov 2004  18:54     using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-CK-1-127-4 (compound J)
Misc Info  : HP5-MS, 30m, 0.25 film, PCI-NH3
```



CI mass spectrum of:

Top: Compound **9** in Soil sample **S/31**, aliquot **CW-1-131-2-S**

Bottom: TMS derivative of the authentic reference standard of **Triethanolamine** [tris(2-trimethylsiloxyethyl)amine] corresponding to compound **9** (MW: **365**)

COMMENTS

1. General

2. Sample preparation

3. Analysis

4. Report

The field length in the Chemstation software often did not allow for the full chemical name and may be truncated or abbreviated.